
# EFFECTS OF PRIOR AGING AT 288 °C IN ARGON ENVIRONMENT ON CREEP RESPONSE OF CARBON FIBER REINFORCED PMR-15 COMPOSITE WITH ±45° FIBER ORIENTATION AT 288 °C

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AFIT/GAE/ENY/09-J02

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## Abstract

The creep and recovery response of carbon fiber reinforced PMR-15 composite with a ±45 fiber orientation was examined in an argon environment at 288ºC. Mechanical testing was executed for unaged specimens as well as specimens aged up to 1000 hours. Tensile tests were performed to determine Young's modulus of elasticity and ultimate tensile strength. The creep tests were carried out at stress levels of 30 and 60 MPa. Creep periods of 25 hours were followed by recovery periods of 50 hours at zero stress. Weight loss measurements as a function of aging time were taken as well. The accumulated creep strain of the unaged specimen was ~.72% at 30 Mpa and ~1.86% at 60 MPa. The accumulated creep strain of the 1000 h aged test specimen was ~.15% at 30 MPa and ~.72% at 60 MPa. Prior aging time reduced the amount of creep strain accumulation, but it did not significantly affect recovery. The carbon fiber reinforced PMR-15 composite had a weight loss of ~1.37% after 500 hours of aging, compared to a weight loss of ~.91% at 250 hours. The specimens experienced an initially rapid weight loss followed by a period of steady increase in weight loss rate.

## Acknowledgments

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# Introduction

## Background

A high performance polymer matrix composite (PMC) typically consists of a thermoset or thermoplastic polymer matrix reinforced with fibers that are much stronger and stiffer than the matrix. The PMCs are lighter, stronger, and stiffer, than conventional materials. Furthermore, composites can be tailored to perform particular functions such as offer high thermal or electrical conductivity, stealth characteristics, the ability to self-heal, communication, and sensor capabilities. The multi-functionality of these materials is perhaps the most important feature. This allows for weight savings, improved fuel economy, increased passenger and payload capacity, and better maneuverability.

Polymer matrix composites (PMCs) were originally developed in the 1960s for use in aerospace and missile systems. These systems demanded a material that could withstand temperatures above 200°C, which was above the operating temperature of most epoxy resins. Research and development led to the use of polyimide polymers as the leading matrix material with high performance carbon fibers as the reinforcement. The high cost of carbon fibers ($400 to $500 per pound) limited their applications to high value military and aerospace systems [23]. Fifty years of research allowed for the use of PMCs to become commonplace through the price reduction of manufacturing as well as confidence in the material's ability to handle an increasing amount of tasks in multiple fields. Commercial aircraft, industrial applications such as pressure vessels, automotive seats and truck beds, and sports and leisure equipment are just a few of the applications for which

PMCs have found a home. The commercial use of PMCs has expanded to such an extent that the Department of Defense only accounts for ten percent of the domestic market and five percent of the world market [23].

The decrease in cost of these materials has allowed for a wider variety of applications within the Department of Defense as well. Today PMCs are used in each of the military services. For example, the US Navy's F/A-18 E/F consists of 19% carbon-epoxy composites by weight [23]. The F-22 Raptor has more than 350 parts made from carbon-epoxy composites, which accounts for 25% of its structural weight, while the Joint Strike Fighter consists of between 25 and 30% composite by weight. The Army's M829A2 antitank weapon uses a composite sabot round that accounts for 385,000 pounds of composite per year. Future roles of composites in the military are expected to increase by hundreds of percent. The Army's Objective Force is a program of advanced future weapons incorporating composites in munitions, armament, and hull structures that will allow the Army to rapidly deploy from a C-130 aircraft. The trend towards faster, lighter, and stronger military systems appears to be tailor made for PMCs.

PMR-15 (Polymerization of Monomeric Reactants-15) is a high temperature polymer developed by the National Air and Space Administration (NASA) Lewis Research Center in the 1970s. The name refers to the process by which it is manufactured while the number designation '15' refers to its average molecular weight of 1500 g/mole of the oligomers [6]. PMR-15 has a combination of attributes that make it ideal for aerospace applications. It has high-thermal stability, good chemical and solvent resistance, as well as an excellent retention of mechanical properties at high temperature. The glass transition

temperature of this polyimide is 348°C. The long-term temperature use of PMR-15 is 288°C [10]. PMR-15 is the most widely used polyimide and therefore the most studied and researched. Carbon fiber PMR-15 composite makes exceptional structural components and is being used in a wide variety of applications such as horizontal and vertical stabilizers, small fuselage sections, wing skins, rotor blades, fuel tanks, and aircraft engine housings [5].

A regrettable feature of PMR-15 is a hazardous material compound called methylenedianiline (MDA). This monomer is a known animal carcinogen, a known liver toxin and a suspected human carcinogen. The danger of this product is great enough to have caused the Occupational Safety and Health Administration (OSHA) and the Environmental Protection Agency (EPA) to heavily regulate the use of MDA. The implementation of these safety measures in handling and disposing of PMR-15 materials causes large financial losses in the industries that use this composite. These safety concerns involving MDA as well as the cost of government regulations has caused certain commercial airlines to completely eliminate PMR-15 from their fleets, despite the advantages in structure, weight, and strength[28].

The search for a replacement of the MDA monomer has focused on finding a substitute that does not contain mutagens while still maintaining the ease of manufacturing, stability, and mechanical properties that are the hallmark of PMR-15. Several competitors have emerged in the last couple of decades. A new class of thermosetting polyimides has developed from these efforts. Dimethylbenzidine (DMBZ) polyimides are used in the autoclave processing developed for PMR-15 to produce T650-35

carbon-fiber-reinforced laminates. The glass transition temperatures of these laminates is 418ºC, which is around fifty degrees higher than PMR-15. This unusually high transition temperature allows for the use of DMBZ-15 in temperatures as high as 343ºC. The weight loss of DMBZ-15 versus PMR-15 was also comparable; .9 versus .7 percent over 1000 hours at 288 C. The mechanical properties of PMR-15 and DMBZ-15 are also similar as can be seen in Figure 1. RP-46, patented in 1991, is a direct PMR-15 replacement

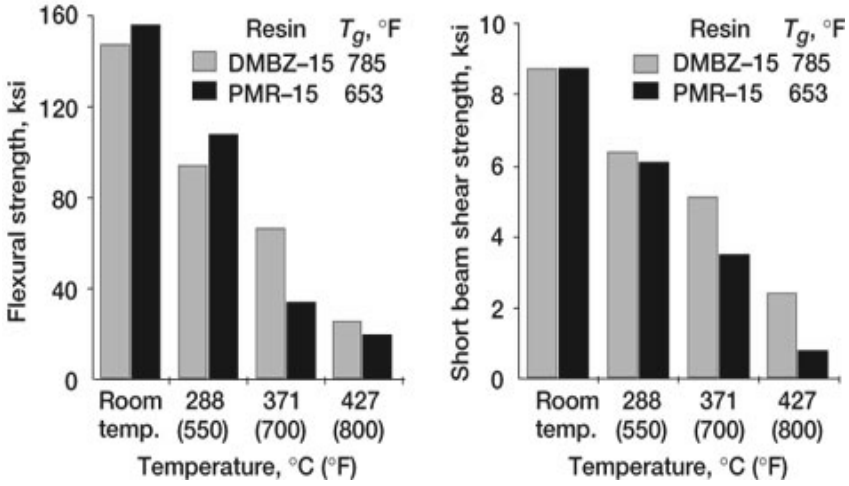


**Figure 1- Comparison of mechanical properties of DMBZ-15 versus PMR-15**

with a very similar chemical makeup. The glass transition temperature of a carbon fiber/RP-46 composite is 397 C which allows for a sustained service temperature of 357 C. The thermo-oxidative stability is very high, which means it resists oxidation that breaks down molecular bonds even at high temperatures.

Prolonged exposure to elevated temperature can affect the mechanical properties of a polymer over time, and thus it is necessary to study these effects independently of oxidation so as to better understand what is physically and chemically happening.

Obtaining this information requires exposing polymers to high temperature in an inert environment prior to mechanical testing. Such testing allows for the degradation of the specimen due to thermal exposure to be studied without the influence of thermal oxidation. The use of PMR-15 requires that engineers and designers have factual evidence of limitations and characteristics so as to utilize the material in a manner consistent with its strengths and weaknesses.

This thesis explores the effects of isothermal aging in an argon environment on creep and recovery behavior of carbon-fiber reinforced PMR-15 neat resin. The results of this research will add to the existing understanding of the time-dependent behavior of this composite.

**Research Objective**

The objective of this research was to explore the effects of prior aging at 288°C in an argon environment on the mechanical behavior, creep, and recovery response of Carbon-Fiber reinforced PMR-15 composite. This research will discuss the effects of prior aging on the Elastic Modulus and creep and recovery response. Weight loss data of the composite was also acquired. The information collected will allow for more accurate predictions of service-life of PMR-15 based components.

**Assumptions/Limitations**

The most important assumption of this thesis is that the oxidation of PMR-15 is negligible at room temperature. The exposure of the specimens is unavoidable as they are exposed immediately after processing and manufacturing. Further exposure to the

specimens occurs when the aging chamber is opened and during weight measurements. This is a common assumption and it has been deemed that the exposure to room temperature will not affect the mechanical properties in any significant way. It was also assumed that the thermal cycles of heating and cooling caused by removal from the aging chamber will not have an adverse effect to the sample's properties. Finally, it was assumed that all testing was done at 288°C in a dry environment so as to avoid consideration of effects caused by moisture absorption.

**Methodology**

This thesis research included several steps. The room temperature elastic modulus was measured for each specimen in order to establish the specimen-to-specimen variability. The next step was to age the specimens for 100, 250, 500, and 1000 hours at 288°C in an argon environment. The final step required subjecting each sample to the creep and recovery test.

**Implications**

The data collected in the course of this research has implications in terms of economics, safety, and engineering. The cost of testing individual components for a specific application is not a viable alternative for any organization. This precludes the use of a material because of the cost of study before implementation. Without studies such as this one, research and development has no data to draw upon in order to understand the uses for which individual materials are feasible. The information gathered through these tests allows for a greater understanding of the limitations of the materials. These limitations are

particularly important for structural materials such as PMR-15 based composites. The engineering uses of composites are expanding. The use of engineered composites is increasing with further advances in aeronautics. Knowledge of how a particular material will respond under any situation is invaluable to designers. This thesis attempts to provide a small portion of that knowledge.

# Literature Review

## Chapter Overview

This section will discuss the chemical makeup and production of PMR-15, followed by a discussion on the general forms of polymer degradation. Literature that is relevant to this thesis research will also be examined. This includes current knowledge of the effect of prior aging at elevated temperature on the mechanical properties of carbon-fiber reinforced PMR-15 composite in an argon environment.

## Carbon PMR-15 Production

PMR (polymerization of monomeric reactants) refers to a method of impregnating carbon fibers with a solution of the monomers, which are then polymerized "in-situ". PMR-15 is assembled from the three monomers shown in figure 2. The suffix 15 refers to the average formulated molecular weight of 1500. This weight is obtained by combining the dimethylester of 3,3',4,4'-benzophenone-tetracarboxylic acid (BTDE), 4,4'-methlyenedianiline (MDA) and monomethyl ester of 5-norbornene-2,3-dicarboxylic acid (NE) in molar ratio of 2:3.087:2. The currently accepted chemical reaction sequence is shown in figure 3.

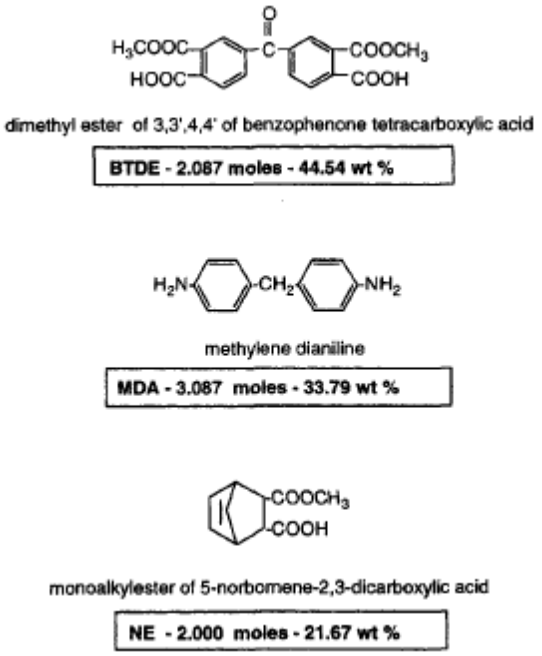


**Figure 2 Stoichiometric ratio of PMR-15 monomers**

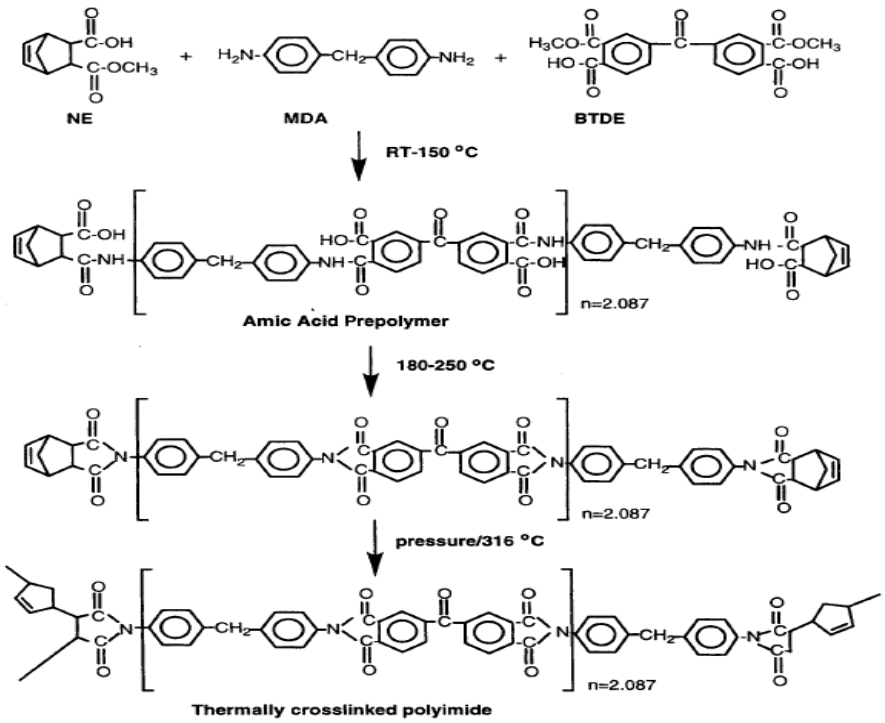


**Figure 3 Sequence of curing reactions for PMR-15**

At 80 to 150°C, condensation of water and methanol between NE and MDA and between MDA and BTDE, causes rapid growth of low molecular weight amic-acid oligomers. Imidization begins in this temperature range as well and continues from 200-250°C, which produces the prepolymer. This prepolymer is used as the prepreg material. Crosslinking reactions begin around 250°C and reach maximum energy at approximately 362°C. During the condensation reactions a considerable amount of water and methanol is produced. The removal of these products is essential to obtaining high quality, void-free, cured composites [22].

Carbon fiber is among the most important fibers for aerospace and military applications. It is engineered for strength and stiffness, but can vary in electrical conductivity, thermal properties, and chemical properties. Physical properties of this fiber are dependent upon the percent of carbon per weight (usually > 92 %), the orientation of the carbon planes, and the degree of crystallization [23]. Carbon-fiber reinforced PMR-15 is produced by impregnating the carbon fibers with the dissolved monomeric solution mentioned above. This induces the monomers to undergo in-situ cyclodehydration. The composite material is formed when polymerization through the nadic endcaps occurs directly on the fiber surface.

**Degradation and Mechanical Behavior Effects**

Different forms of degradation have specific effects on the specimens of this thesis. Full understanding of polymer degradation theories will ultimately allow for greater insight into the mechanical and chemical changes taking place within the polymer.

Physical aging is a process by which the material rearranges itself into a lower energy state via a thermodynamically reversible volumetric response. The glass transition temperature of a material determines whether or not physical aging is initiated. When a material is below the glass transition temperature the material is not in equilibrium and so strives to attain a thermodynamic equilibrium state. This process can be accelerated with higher temperatures and is the reason that the specimens in this thesis are aged at elevated temperatures. Increased yield stress and modulus, decreased fracture energy, and decreased toughness and elongation are a few of the effects common to this process. Research has also yielded the result that physical aging may have an effect on the time-dependent mechanical properties and the rate dependent failure processes [33].

Chemical aging is different than physical aging in that it is a nonreversible volumetric response. This process involves the rearranging or breaking of chemical bonds within the polymer. Chain scission reactions, increased or decreased crosslink density and hydrolysis are a few of the responses included within this type of aging. Mechanical properties such as modulus, elongation, or toughness can change quite drastically due to these reactions. Common effects of chemical aging include lower molecular weight, etching of the surface, discoloration, voids, and hardening [6].

The final type of polymer aging is Oxidation. This process is the reaction of polymer chains to an oxygen environment. This response is accelerated by elevated temperatures and leads to lower molecular weight, a lower glass transition temperature, embrittlement, additional crosslinking, and changes in mechanical properties like modulus, elongation, and toughness.

High temperature polymers are usually employed in environments where all of these types of aging are occurring simultaneously. The changes caused by the individual processes need to be better understood so as to allow more accurate modeling of the responses of the materials. Typically the net effect of degradation is the only information available. The degradation caused by oxidation in this study is negligible due to the nature of the argon testing environment.

**Thermal Stability and Degradation**

An isothermal stability study was conducted at the NASA Glenn Research Center in 2001 by Bowles et al [3]. This testing was completed in order to address the need for the development of a reliable model to describe the effects of elevated temperature isothermal aging of polymer matrix composites. The specimens were exposed to multiple temperatures (204, 260, 288, and 316°C) in oxygen and nitrogen. The composite of interest in this study was T650-35/PMR-15 composite (24 by 23, 8 harness satin weave graphite fiber fabric). The quality of the materials was ensured via nondestructive evaluation procedures and by metallographic photography during aging. The specimens were machined to size by a water-cooled micromachining diamond saw. All specimens were dried 24 hours at 125°C prior to testing to ensure dry conditions. Specimens were kept in a desicator after aging to minimize moisture absorption. The glass transition temperature was recorded by dynamical mechanical analysis (DMA) testing. The two components of the complex shear modulus were measured as a function of temperature. This allowed the glass transition temperature to be determined by measuring the intersection of the two tangents to the linear portions of the stored shear modulus G curve

where it suddenly dropped to a minimum. Figure 4 shows the storage moduli of the T650-35/PMR-15 composite specimens.

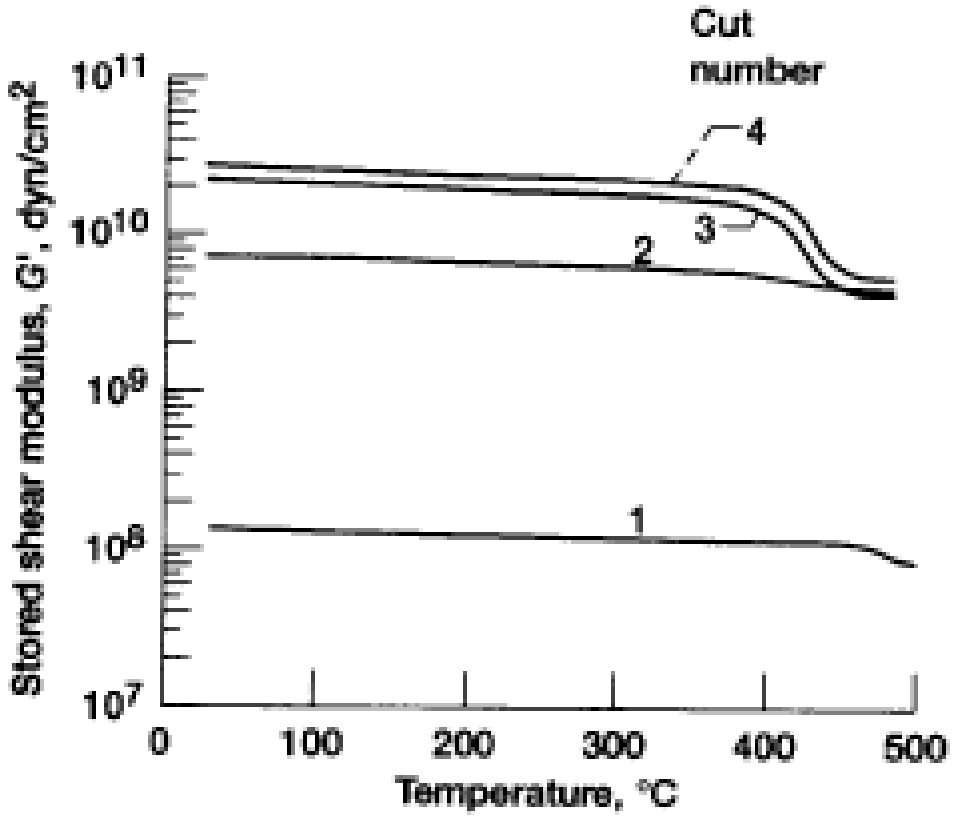


**Figure 4 Stored moduli of T650-35/PMR-15 composite. Figure from Ref [3].**

Note that in Figure 5 it can be seen that the density of the specimen is decreasing with increased aging time. This is due to the release of gaseous products from the material. This response varies from when samples are aged in air as can be seen in Figure 6. The increase in density seen in the oxygen environment specimens is caused by the absorption of oxygen. This process increases in rate as more and more cracks are formed in the material which allows for a larger surface area and therefore more oxidation.

| Sample number | Aging temperature, °C | Aging time, hr | Density, ρ, g/cm³ | Fiber, wt % |
|---|---|---|---|---|
| 1 | 316 | 500 | 1.530 | 64.11 |
| 2 | | | 1.535 | 64.80 |
| 3 | | | 1.506 | 64.22 |
| 4 | 316 | 500 | 1.535 | 68.51 |
| 5 | | | 1.556 | 66.96 |
| 6 | | | 1.558 | 67.98 |
| 7 | Unaged | Unaged | 1.584 | 67.89 |
| 8 | | | 1.578 | 67.09 |
| 9 | | | 1.571 | 67.99 |
| 10 | Unaged | Unaged | 1.588 | 68.84 |
| 11 | | | 1.595 | 68.28 |
| 12 | | | 1.588 | 69.22 |

**Figure 5 Densities and fiber contents of T650-35/PMR-15 composite. Figure from Ref [3].**

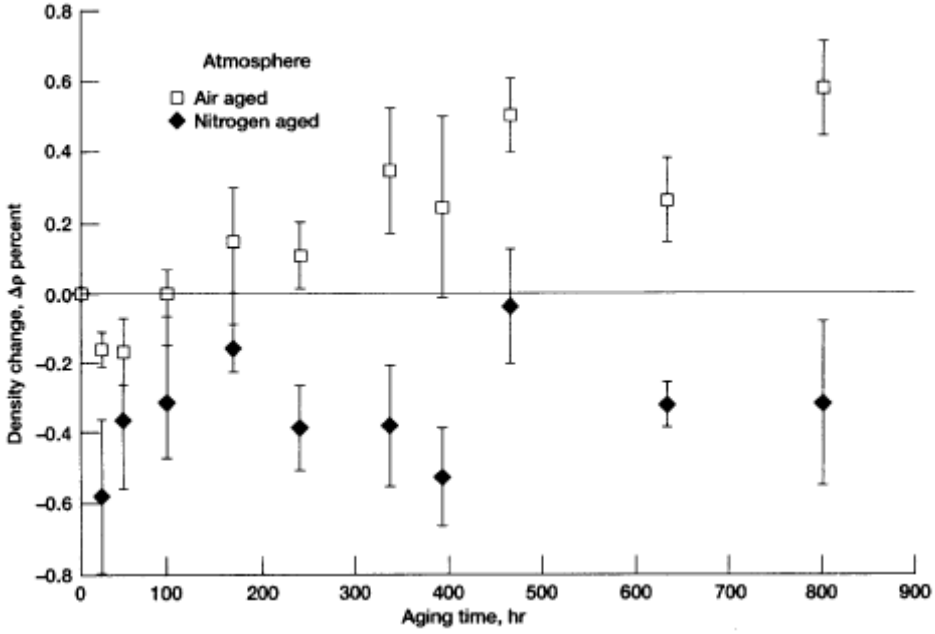


**Figure 6 Density change as a function of aging time. Figure from Ref [3].**

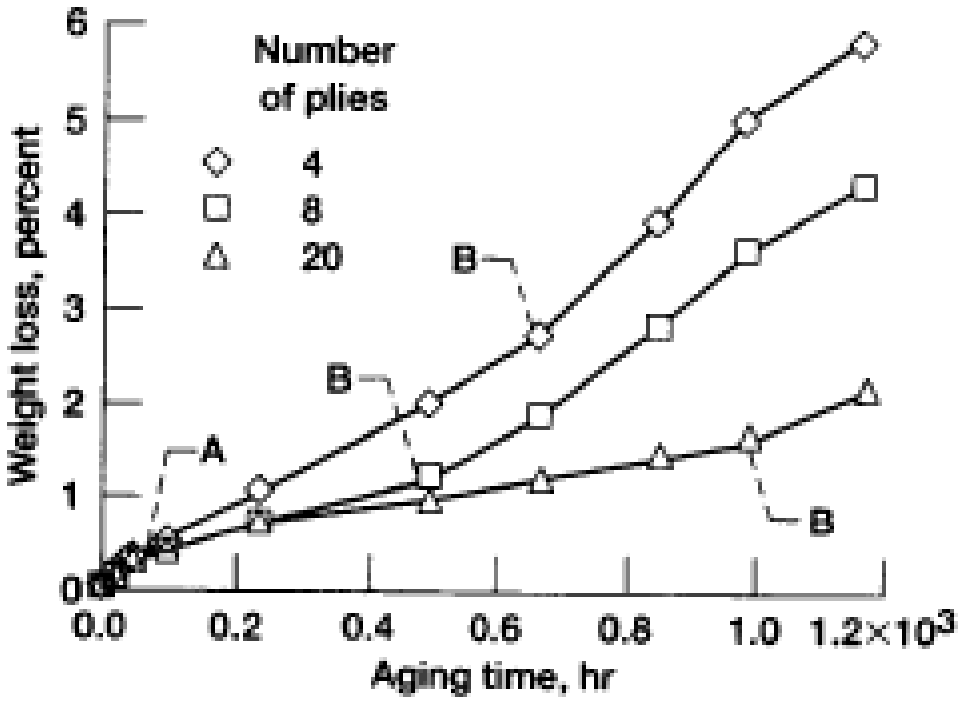


**Figure 7 Weight loss percent as a function of aging time for T650-35/PMR-15 composite at 316°C. Figure from Ref [3].**

The thermal degradation of a material can be tracked through the specimen's weight loss. This weight loss is explained by the vaporization of low molecular weight molecules released through chain scission. For weight changes in oxygen the measurements must be normalized by the surface area as the oxidation process is primarily on the surface. Inert environments require normalization by the specimen volume, because the degradation is occurring throughout the specimen.

Schoeppner et. al. [32] evaluated unidirectional G30-500/PMR-15 composite specimens aged in air and argon at elevated temperatures in order to study the weight, surface area, and volume changes that occur. The specimens were machined using a diamond wet-saw with distilled water as a cooling medium. Specimens were placed in a

vacuum oven for at least 48 hours at a temperature of 105°C to ensure low moisture content. Four different geometries were cut from the panel in order to obtain four different surface area ratios. Two other specimens were cut from the panel in order to monitor the propagation rate of oxidation along the length of the fibers as well as transverse to the fibers. Specimens were then stored in a nitrogen purged desiccators prior to aging. In order to isolate the effects of oxidation on material weight loss, several specimens were aged in argon at 288°C. This allowed the effects of pure thermal degradation to be isolated as well.

A scanning electron microscope (SEM) was used to examine the damage caused to the specimens. Fiber-matrix interphase degradation was observed and explained as the result of localized micromechanical residual stressed caused by the mismatch in the coefficient of thermal expansion between the matrix and the fibers. The local stoichiometry of the polymer may be altered in the fiber-matrix interphase by the presence of glass fiber reinforced coupling agents as well. This could cause preferential oxidation along the fiber [32].

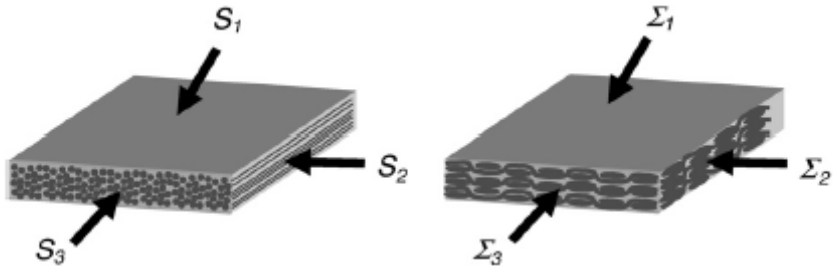


**Figure 8 Surface area designators for unidirectional and woven composites. Figure from Ref [32]**

| Specimen | $S_1$ ($mm^2$) | $S_2$ ($mm^2$) | $S_3$ ($mm^2$) | $S_1 + S_2 + S_3$ ($mm^2$) | $(S_1 + S_2)/S_3$ |
|---|---|---|---|---|---|
| R1 | 571.0 | 37.3 | 186.3 | 794.6 | 3.27 |
| R2 | 616.0 | 85.8 | 85.5 | 787.3 | 8.21 |
| R3 | 1258.2 | 119.7 | 119.7 | 1497.6 | 11.51 |
| R4 | 572.5 | 203.9 | 40.9 | 817.3 | 18.98 |

**Figure 9 Surface areas and surface area ratios for four specimen geometries**

The dimensional changes observed were measured using a standard notation that can be seen in Figure 8. The values of the four different geometry ratios can be seen in Figure 9. The three different types of unidirectional composite surfaces are defined as $S_1$ - the area of the non-machined resin-rich surface, $S_2$ - the area cut parallel to the fibers, and $S_3$ - the area cut perpendicular to the fibers. Woven composites have three surfaces as well. $\Sigma_1$ is the area of the non-machined resin-rich surfaces, $\Sigma_2$ is the surface area cut perpendicular to the warp fibers, and $\Sigma_3$ is the surface area cut perpendicular to the fill fibers.

The volume change for the specimens aged in argon was found to be consistently lower than the volume change of the specimens aged in air. Non-oxidative degradation, as Bowles et. al. [3] also believed, was likely caused by degassing of low molecular weight components created by chain cleavage in the polymer. As discussed earlier, the degradation of specimens aged in argon was expected to be a function of volume, not surface area. Figure 10 shows the volume change for one type of geometry in air and in argon. The variation of volume between the separate geometries was not significant.

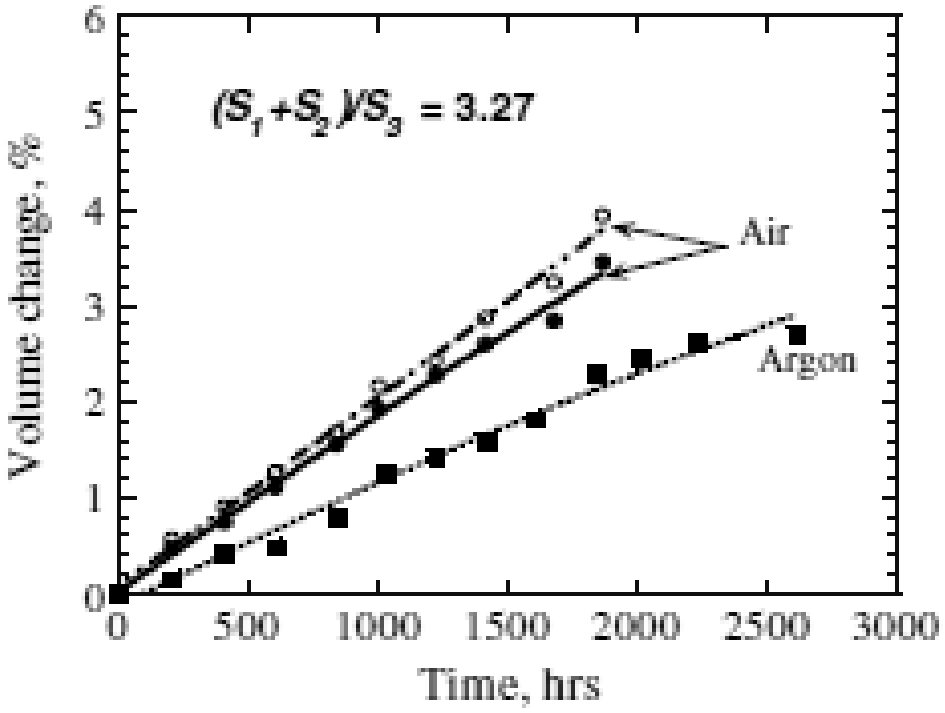


**Figure 10 Volume changes for specimens aged in air and in argon at 288C. Figure from Ref [32]**

The accepted method of quantifying the thermal oxidative stability of high-temperature polymer matrix composites is to examine the weight loss as a function of isothermal aging time. The weight loss of the specimens was measured for the air and

argon environments at a temperature 288°C. An example of the results can be seen in Figure 11. The comparison of the four different geometries yielded the result that weight loss was independent of geometry. The volume of the specimen was necessary to normalize the results in order to allow understandable comparisons between specimens. This is due to the fact that the thermal aging in an inert environment is a volumetric response [32]. The air aged specimens showed an opposite result, with the weight loss proving to be dependent upon surface area.

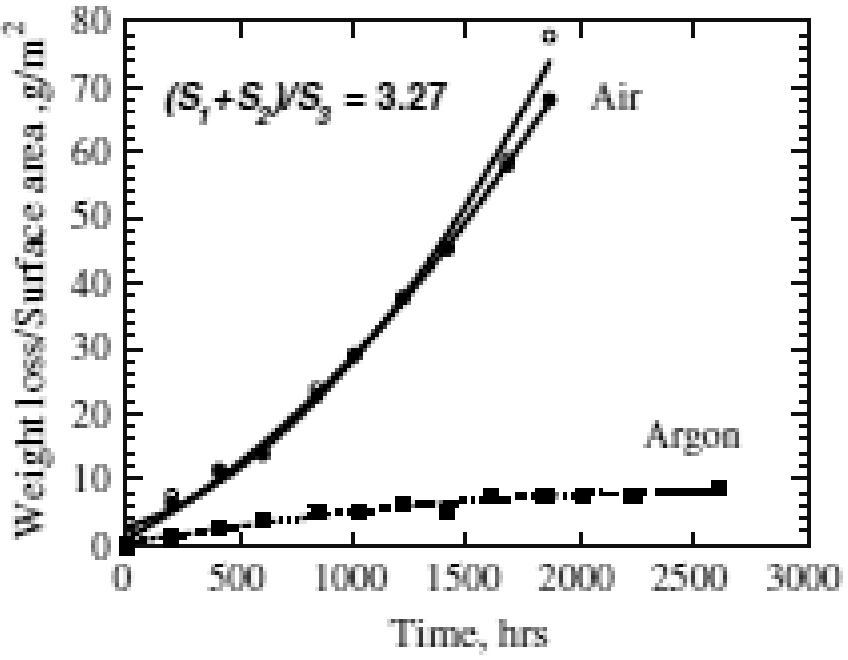


**Figure 11 Normalized weight loss for PMR-15 composite aged in air and argon at 288°C. Figure from Ref [32].**

**Summary**

This was a brief summary of the current and ongoing research on the mechanical and chemical effects of carbon fiber reinforced PMR-15 composite. The focus of this section was to illustrate the effects that an argon environment will have upon a specimen

subjected to elevated temperatures. Long-term exposure to high temperatures and an oxidizing environment is understood to cause degradation to materials. The effort to classify the effects observed from such an environment into neatly defined categories is an ongoing endeavor.

# Material and Specimen

## Overview

The following section discusses the manufacturing, geometry, aging, and preparation of the Carbon fiber reinforced PMR-15 specimens.

## CYCOM 2237/PMR-15

The test material of composite was provided by HyComp Incorporated (Cleveland, Ohio). It is formulated for high temperature applications where low void content is critical. The more attractive features include high strength and dimensional stability at elevated temperatures, excellent handling characteristics, good product consistency, and microcrack resistance. In the 260°C (500°F) to 288°C (550°F) range, this material has the best overall balance of processing behavior, thermo-oxidative stability and retention of properties. A glass transition temperature of 338°C (640°F) makes this product especially useful in aerospace applications. Specifically, carbon-fiber reinforced PMR-15 is ideal for jet engines and other components such as bypass ducts, gear box covers, fan stator and vane assemblies, nozzle flaps, and compression molded bearings.

## *Specimen Fabrication*

### Material Processing

The fabrication of this material is a very precise process. The variability of mechanical properties is highly dependent upon the processing cycle. The history of this panel is unknown prior to its acquisition by the Air Force Institute of Technology. However, measuring the initial elastic modulus of each specimen, specimen-to-specimen

variability can be assessed. The PMR-15 matrix is reinforced with T650-35 carbon fibers woven in an 8-harness satin weave with a ±45 fiber orientation. The initial cure was an autoclave laminate cure cycle followed by a free-standing post cure cycle. Typical properties of the T650-35 carbon fiber fabric are shown in table 1. Details of the laminate cure cycle and the post-cure cycle are shown in tables 2 and 3, respectively.

| **T650-35 carbon fiber 8-harness satin fabric reinforced** | | |
|---|---|---|
| **Material Property** | **RT** | **550°F (288°C)** |
| Tg by TMA-Exp, dry °F (°C) | 627 (330) | — |
| Warp tension, [0] | | |
| * Strength, ksi (MPa) | 138 (951) | 121 (834) |
| * Modulus, msi (GPa) | 10.4 (71.7) | 11.1 (76.5) |
| Warp compression, [0] | | |
| * Strength, ksi (MPa) | 145 (999) | 119 (820) |
| Modulus, msi (GPa) | 10.6 (73.1) | 12.0 (82.7) |
| Fill compression, [0] | | |
| Strength, ksi (MPa) | 104 (717) | 88 (606) |
| Modulus, msi (GPa) | 9.3 (64.1) | 9.7 (66.8) |
| In-plane shear, [(+45/-45)] | | |
| Strength, ksi (MPa) | — | 8.8 (60.7) |
| In-plane shear, Iosipescu | | |
| Strength, ksi (MPa) | 11.6 (79.9) | 5.9 (40.6) |
| Modulus, msi (GPa) | 0.68 (4.6) | 0.43 (2.9) |
| Apparent interlaminar | | |
| Strength, ksi (MPa) | 8.4 (57.9) | 7.0 (48.2) |
| Compression interlaminar | | |
| Strength, ksi (MPa) | 7.5 (51.7) | 5.7 (39.3) |

**Table 1 T650-35 Carbon Fiber 8 harness satin fabric material properties**

| Laminate cure cycle |
|---|
| Cure laminates in an autoclave as follows: |
| 1. Apply 2-6 inHg vacuum |
| 2. Raise temperature to 425±10°F at a rate of 0.5 – 3.0°F per minute. |
| 3. At 300±10°F apply full vacuum. |
| 4. Hold for 180 – 210 minutes at 425±10°F (temperature based on |
| 5. Then raise temperature to 475±10°F at a rate of 0.5 – 3.0°F per |
| 6. At 450 – 475°F apply 200±10 psi pressure at a rate of 15 – 20 psi per |
| 7. Hold @ 475±10°F for 30 – 55 minutes. |
| 8. After hold period, raise temperature to 600±10°F at a rate of 0.5 – |
| 9. Hold for 180 – 210 minutes at 600±10°F (temperature based on |
| 10. At the end of hold, cool slowly under pressure. |
| 11. When temperature is below 400°F, release pressure and vent |
| 12. Cool to 160°F prior to removing from autoclave. |

**Table 2 Laminate Cure Cycle**

| Post-cure (free-standing) |
|---|
| 1. Post-cure in an oven starting at room temperature to 600±10°F @ 10 |
| 2. Hold @ 550±10°F for 5 hours. |
| 3. Then raise temperature to 600±10°F at a rate of 0.5 – 1.0°F per minute |
| 4. Hold @ 600±10°F for 10 hours. |
| 5. Cool to 160°F prior to removing from oven. |

**Table 3 Post-cure Cycle**

**Specimen Manufacture and Geometry**

Standard dog bone shaped test specimens were utilized in this study. The test specimen is shown in Figure 12. This shape ensured failure in the gage section during the tensile tests. The specimens were cut from the composite panel by the Air Force Institute of Technology machine shop using a diamond saw. After machining all specimens were washed with common household soap to remove any contamination caused by the machining process. The specimens were then placed in a vacuum oven at 105°C for 4 days to remove any moisture absorbed during post cure. All specimens were stored in a dry-air-purged desicator before and after testing.

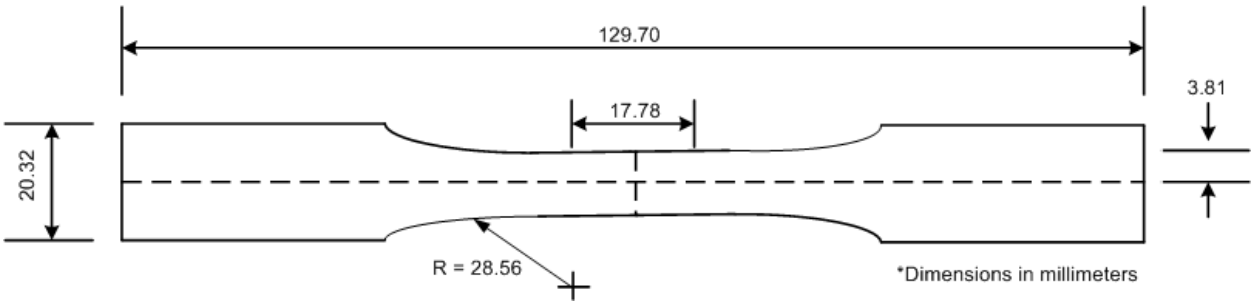


**Figure 12 Dog bone specimen dimensions**

**Specimen Tabbing and Dimpling**

Fiber glass composite tabs were attached to the gripping sections of the test specimens using M-Bond 200, a room temperature epoxy adhesive, in order to protect the specimens from the rough surface of the grip wedges. The mechanical testing conducted in this study required the use of a high temperature extensometer. In order to ensure accuracy, small indentations, or "dimples", were created in the specimens to provide continuous contact for the extensometer probes.

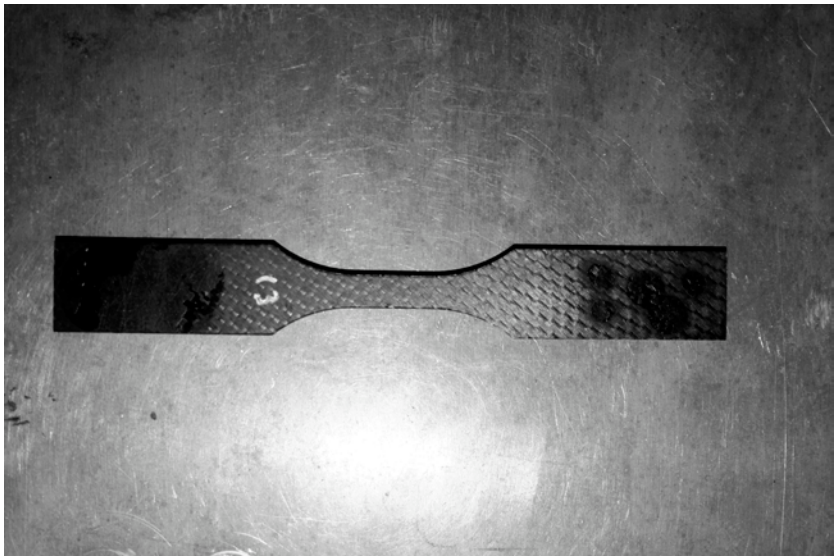

**Figure 13 Test specimen before tabbing**

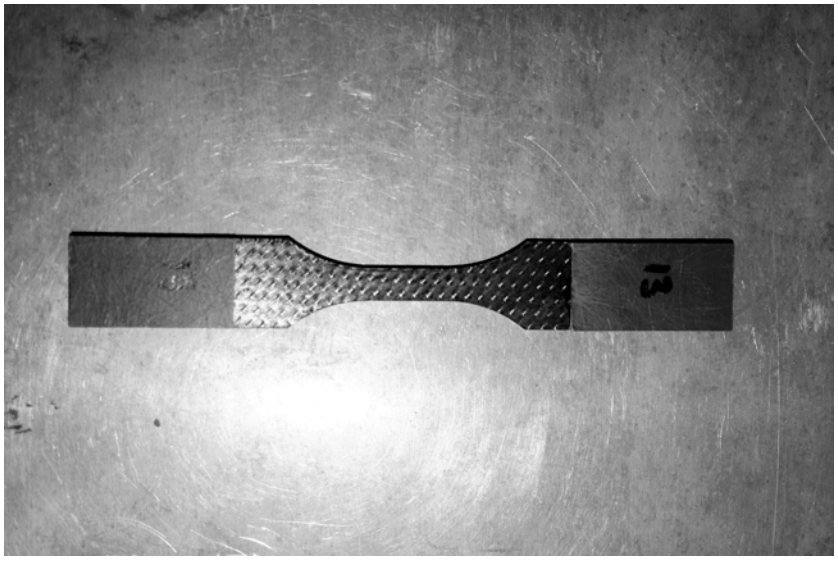

**Figure 14 1000 hour aged test specimen after tabbing**

**Aging**

The specimens were aged in an ultra high purity argon gas (99.999% pure) at 288°C for varying periods of time ranging from 100 to 1000 hours. The aging was accomplished in a Blue M Oven, model 7780. All specimens were placed on a quartz cloth to ensure no contamination from the oven trays. The argon gas was supplied to the oven at a positive pressure to ensure no contamination by air. The flow rate of the argon was approximately 30 SCFH during the steady state operation and 150 SCFH during the purge cycle. The removal of specimens at pre-specified times caused the oven to automatically purge itself for 18 minutes in order to ensure a pure argon environment. All specimens were removed without cooling. It was assumed that the opening of the oven did not introduce enough moisture and/or air to cause significant oxidation or hygrothermal effects. Immediately upon removal from the oven, all specimens were stored in a dry air-purged desicator prior to mechanical testing. The desicator was kept at a constant relative humidity of less than

10% to ensure the absence of moisture effects. This allowed all mechanical properties to be considered “dry” mechanical properties.

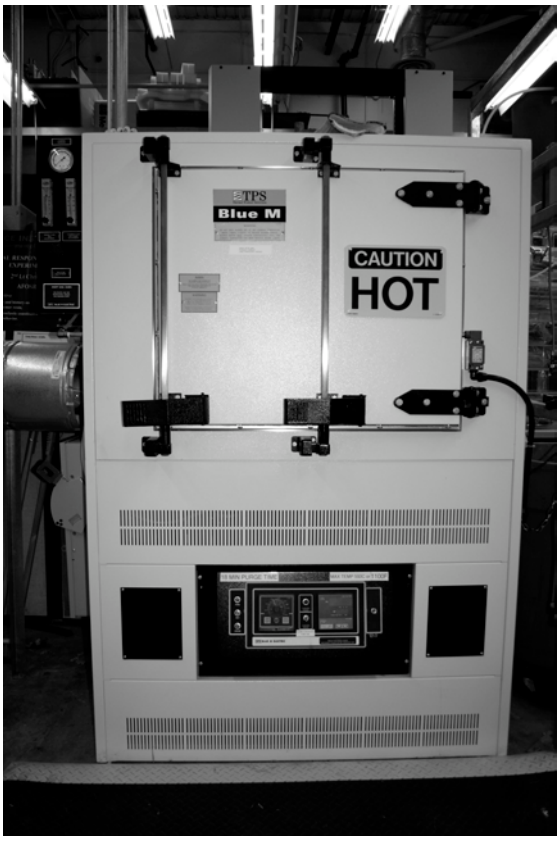


**Figure 15 Aging Chamber**

**Relative Humidity**

The relative humidity of the ambient air during mechanical testing is assumed to be of no consequence in terms of its effects on the mechanical properties of the test specimens. This is a viable assumption as can be seen from previous research which shows that even in a very humid environment (RH> 70%) the relative humidity around the specimen is very low (RH< 1%) due to the high temperature of the test (288°C) [6]. This ensures that no moisture effects were introduced during testing.

# Experimental Setup and Testing Procedures

## Chapter Overview

The following section examines in detail the test apparatus and the testing procedures necessary for examining the creep response of Carbon Fiber reinforced PMR-15 composite.

### *Test Equipment*

## Servo Hydraulic Machine

Mechanical Testing of the specimens required the use of a MTS servo-hydraulic model 810 with a 3 KIP load cell. This machine was vertically configured with a MTS model 318.10 load unit and equipped with the TestStar II digital controller for input and data acquisition. The temperature of the specimen was controlled by a MTS single zone furnace model 653.01A outfitted with a MTS model 409.83 temperature controller. This machine had the MTS 647.02B wedge grips, which were capable of applying a grip strength up to 21 MPa (3000 psi). Circulating water during high temperature testing cooled the grips.

The wedge grip strength used in all testing was approximately 4.8 MPa. Each specimen was heated at a rate of 2ºC/min and kept at the target temperature for a dwell period of 30 minutes prior to testing to avoid the effects of temperature overshoot.

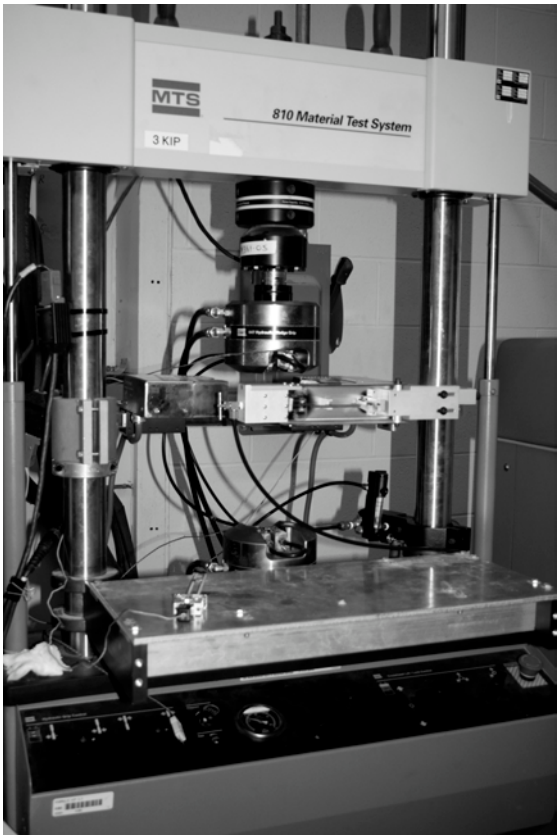


**Figure 16 3 KIP MTS hydraulic machine**

**Cooling System**

During High temperature testing it was required to keep certain apparatus within their operating temperature. As mentioned previously the wedge grips required cooling, which was provided by a NESLAB model HX-75 chilled water system. This system kept the grips inside the operating temperature of -18°C to 65°C with the use of 9°C to 24°C water. Another piece of equipment affected by the high temperature was the extensometer. This device required air-cooling as well as a heat shield to keep thermal effects to a minimum.

**Extensometer**

The strain measurement of each specimen was obtained with the use of a MTS model 632.53E-14 axial extensometer. This piece of equipment has a measurement range of -10% to +20%, and has two 3.5 mm diameter alumina extension rods that are placed in the dimples of each specimen. They are held in place via spring with a 3 N contact force so as to not slip during testing. The alumina rods were each 150 mm long, which allowed for minimal thermal affects by the furnace. The maximum operating temperature of this device is 1200°C with air cooling and 650°C without air cooling.

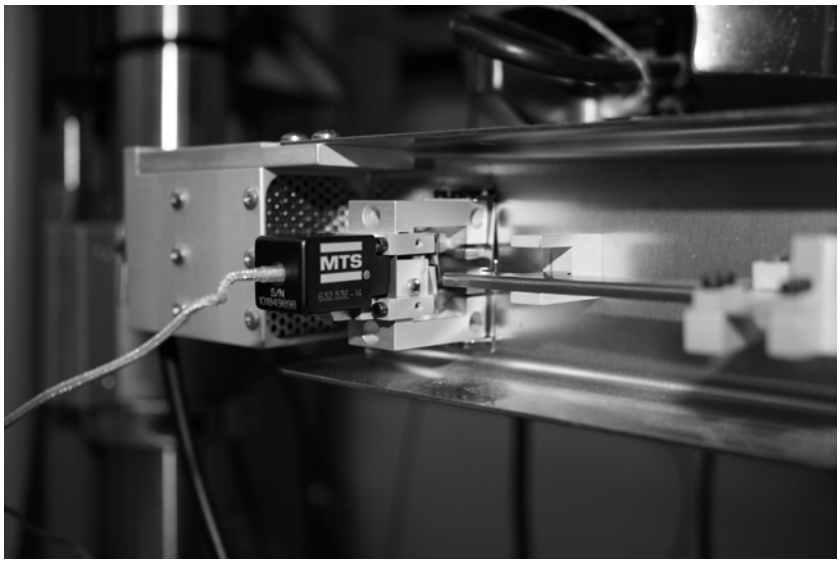


**Figure 17 MTS High temperature axial extensometer assembly with heat shield**

**Figure 18 MTS High temperature axial extensometer attached to test specimen**

## Computer Software

The software in this thesis included the use of MTS TestStar II Digital Controller for all input signal generation and data acquisition and Windows Excel for data processing. TestStar has programs such as MTS Station Builder, which allows the user to construct a configuration file for specific types of materials. The MTS Station Manager application allows the user to manually control specific functions of the MTS 3 KIP machine such as force, displacement, and temperature. This program also gives the user direct manual control of features such as hydraulic strength, interlocks on station parameters, and monitoring real time input and output signals. The MultiPurpose Testware (MPT) allows the user to create test procedures such as the creep and recovery test as shown in the figure. This program permits the user to construct automated testing and data acquisition, which is very convenient for long-term testing.

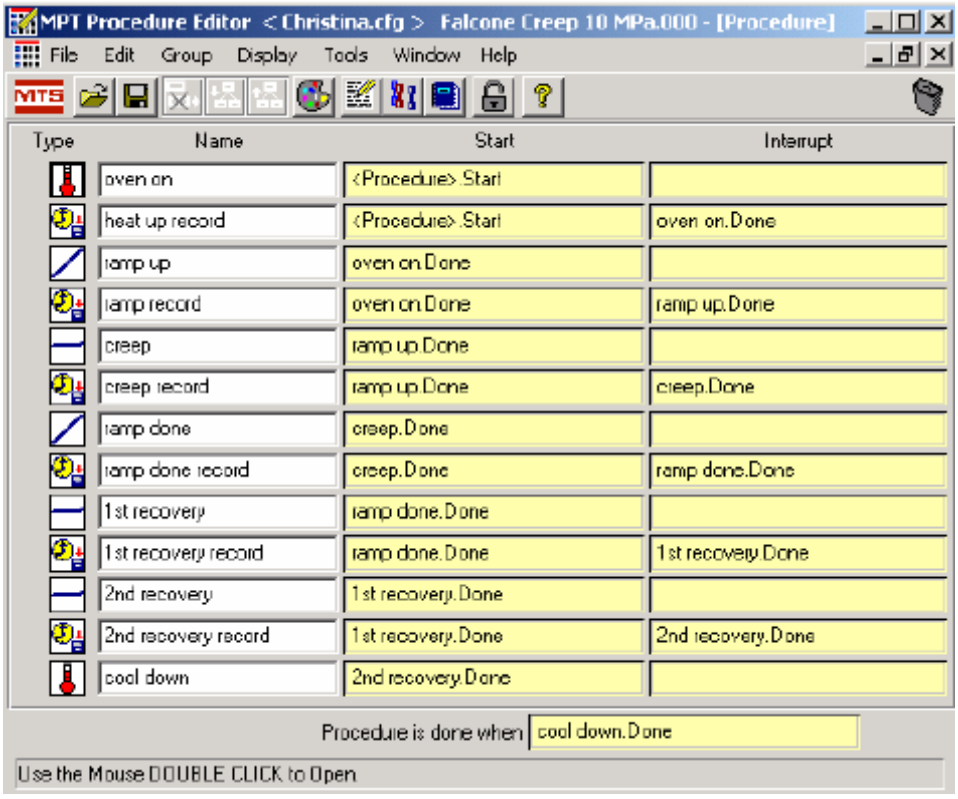


**Figure 19 MPT Creep Test Procedure. Figure from Ref. [10]**

**Furnace Equipment**

The high temperatures required for the specimen mechanical testing was obtained through the use of a MTS 653 Furnace. The furnace is designed as a two piece unit that slides together to enclose the specimen during testing. This allows the furnace to be removed during specimen and extensometer setup. This furnace has an operating temperature of 100°C to 1400°C. Each half of the furnace has a silicon carbide heating element wrapped in silica foam as an insulator. The insulation was filed down to allow the specimen gripping sections space to pass through as well as the extensometer probes access to the specimen during testing. The furnace was controlled via the MTS 409.83 Temperature controller as shown in the figure. The feedback loop between the S-type

thermocouple integral to the furnace and the controller allowed the temperature to stay within required temperatures. The variation between the halves of the furnace unit during testing did not exceed 7°C.


**Figure 20 Temperature controller**


**Figure 21 Furnace**

***Experimental Procedures***

**Determining Test Temperatures**

A temperature calibration was necessary to determine the correct temperature that the controller needed to be set at in order to obtain a testing temperature of 288°C. That temperature, 290°C, was found by attaching two R-type thermocouples to the gage section of a specimen and then raising the temperature manually at a rate of 1°C/min until the target temperature of 288°C was attained. The thermocouples were attached with Kapton tape and wire to each side of the specimen to obtain accurate temperature readings on both sides of the furnace. This process was repeated until a variation between the two sides was less than 6°C. The specimen was then allowed to dwell at that temperature for 10 hours to ensure accurate measurements. The thermocouples did not vary by more than 5°C during the dwell period. The R-type thermocouples were monitored using an Omega OMNI-CAL-SA-110 two channel temperature sensor.

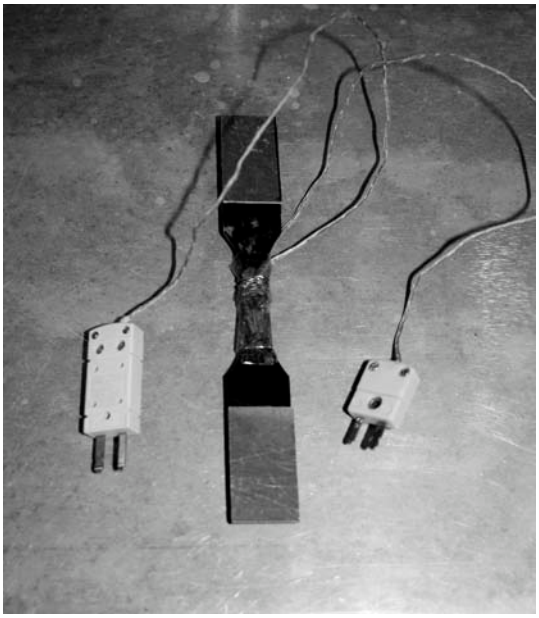

**Figure 22 Temperature calibration specimen with two attached R-type thermocouples**

**Test Procedure**

Preparatory steps were required before any testing could be accomplished. The first step in every experiment was to warm up the hydraulics of the MTS machine. This was accomplished by means of the Function Generator application, which cycled the actuator in displacement control for thirty minutes. The individual specimen geometry was needed in order to calculate the correct force to use for the test. The average thickness and width of each specimen was used to obtain the cross-sectional area of the gage section. This was in turn used in conjunction with the desired stress level to find the force. Dimples were added to each specimen to counter extensometer slippage.

Every test required mounting a specimen. The first step in that process was to put the MTS machine in displacement control and place the grips so that the specimen fit

properly. The specimen was centered in the machine and then the top grips were closed. The load cell was switched to force control and the load cell was zeroed. The bottom grips were then quickly closed as well. The extensometer rods were then placed in the dimples of the specimen. The furnace was closed around the specimen and after ensuring that the extensometer had not been moved, the strain displacement was zeroed. The interlocks, which turn off hydraulic power to the machine, were set so as to prohibit negative strain or compression of the specimen. During testing the machine was kept in force control at zero so that thermal effects would not cause any stresses. Immediately after heat up, a dwell period of thirty minutes ensured thermal equilibrium in the specimen had been reached.

## *Test Descriptions*

### Initial Elastic Modulus Measurements

Standardized processing usually ensures common material properties. The nature of Polymer Matrix Composites allows for variations within individual panels that are not common to other materials. The specimen-to-specimen variation data is required in order to ensure accurate results. This was accomplished with an initial room temperature elastic modulus test of each specimen after machining and prior to aging. The tests were carried out in the laboratory air environment. Each sample was loaded to 3 MPa at a rate of 1 MPa/s and unloaded to zero at the same rate for 3 cycles. This was well below the permanent strain threshold for this material to ensure a linear response. The variation in the specimens was not significant.

**Monotonic Tensile Test**

Tensile tests to failure were conducted for each of the aged and unaged groups. These tests were conducted at an elevated temperature of 288°C. The data obtained from these tests provided Young's Modulus of elasticity and the Ultimate tensile Strength (UTS). Prior research has pointed out the possibility that test data may have been corrupted by the dimples in each specimen [1].

**Creep Tests**

Creep and Recovery tests were conducted on all aging groups and on unaged specimens as well. Previous research on Carbon Fiber reinforced PMR-15 composite established the proper creep stress levels of 30 MPa and 60 MPa for the specimens [7]. The creep period for the samples was 25 hours with a recovery period of 50 hours at zero stress. The specimens were loaded and unloaded at a rate of 1 MPa/s in all tests. Data was collected during all of the stages of the test; heat, load-up, creep, unload, and recovery. Obtaining the proper amount of data in a test is arguably one of the most important aspects of material science. To ensure proper amounts of data collection, the amount of data taken from each test varied according to need. The load and unload data was taken with very little time in between data points. The creep and recovery data series had larger time intervals so as to avoid excess. Within the recovery period, data was taken every second for the first 10 minutes, every 10 seconds for the next hour, and every minute for the remaining time. This was due to the fact that most recovery happens relatively quickly after unloading. The recovery period was followed by a cool down to room temperature. No specimen failures occurred during testing.

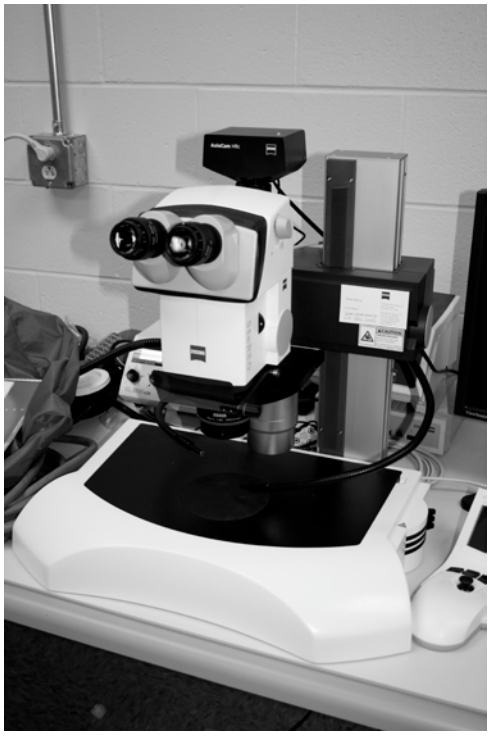

**Figure 23 ZEISS Discovery. V12 Model Stereo Microscope**

## Weight Measurements

Each group of specimens included a rectangular blank sample for the purpose of measuring weight loss as a function of aging time and environment. The initial weight of all the blanks was taken after washing. All weight measurements were taken with a Metler Toledo AG245 Microbalance with a resolution of 0.0001 grams. To maintain near zero moisture the samples were stored in a dry-air-purged desicator before and after aging. To examine the damage done to the specimens, a section of each specimen was cut, mounted, and polished in order that the samples could be viewed via a digital microscope. The specimens were mounted in a room temperature cured epoxy, sanded with 320 and 600 grit sandpaper, and polished with .03 micron silica slurry by a Buehler PowerPro 5000 variable speed grinder-polisher. The micrographs obtained can be found in Appendix A.


**Figure 24 Metler Toledo AG245 Microbalance**


**Figure 25 Buehler PowerPRo 5000 Variable Speed Grinder-Polisher**

**Summary**

Room Temperature Elastic Modulus tests were conducted on all specimens to access variability within the specimen panel. Samples were aged in argon for different durations at an elevated temperature of 288°C. Weight measurements were taken at different times to analyze weight loss versus exposure. Creep tests were performed on all aging groups for 25 hours at creep stress levels of 30 MPa and 60 MPa followed by recovery for 50 hours.

## Results and Discussion

### Overview

This section documents and analyzes the results obtained from this thesis research. The data from each experiment is examined in detail and associated to theory where appropriate.

### Weight Loss Measurements

The weight of two rectangular specimens aged in argon at 288°C was measured as a function of aging time. The weight loss of the specimens was due to thermal degradation. Thermo-Oxidative effects were not present in the specimens due to the argon environment. An argon environment means that the weight loss is dependent upon the sample volume as the entire sample is releasing various products during the aging process. The weight loss factor is a standard of measurement which allows samples of different geometries and sizes to be compared. It is obtained by normalizing the weight loss by the volume of the sample. All weight measurements were taken with a Metler Toledo AG245 microbalance with a resolution of .0001 grams. All samples were stored in a dry-air –purged desicator to maintain a near zero moisture content. Volume measurements were taken before aging. Figure 26 shows the weight loss factor as a function of aging time for the two rectangular specimens.

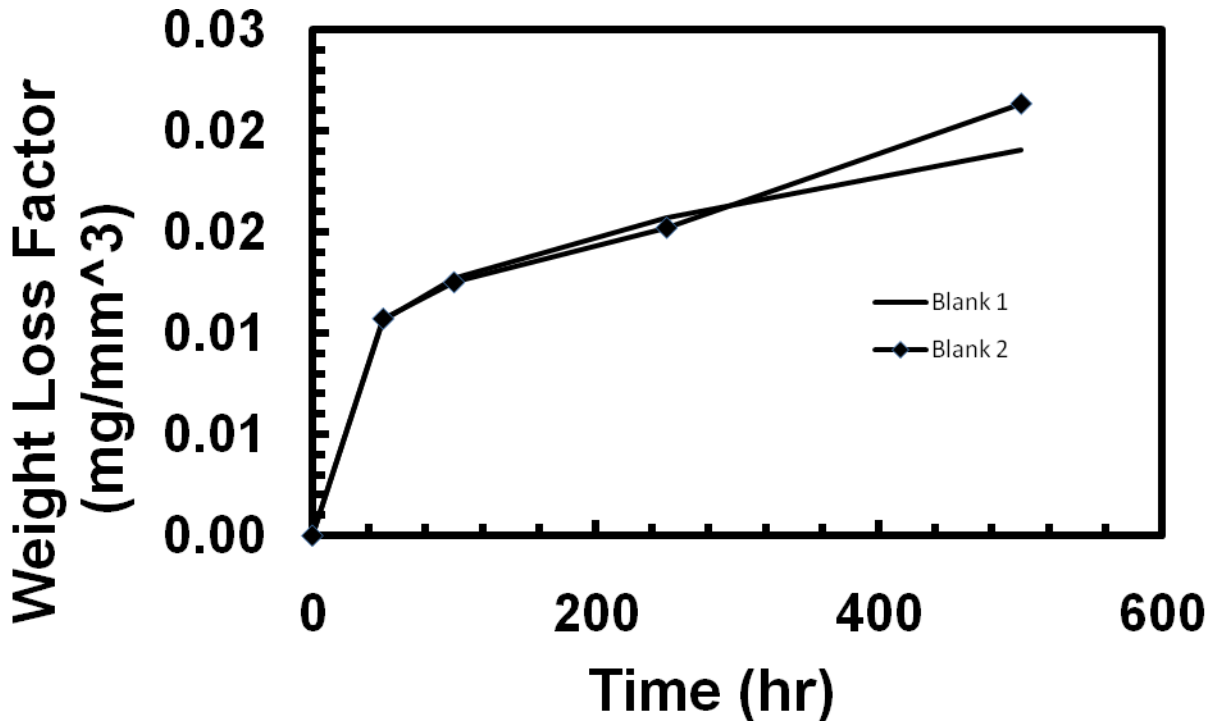


**Figure 26 Weight loss factor of two rectangular specimens as a function of aging time for carbon fiber reinforced PMR-15 composite at 288°C in argon**

These values are close to previous experiments, indicating that the study performed as expected [15]. It is possible to see that there is a rapid weight loss initially followed by a period of steady increase in weight loss rate. This is attributed to the loss of low molecular weight particles as the polymer is degraded by the temperature over time [3]. Figure 27 shows the percent of weight loss as a function of aging time for both specimens.

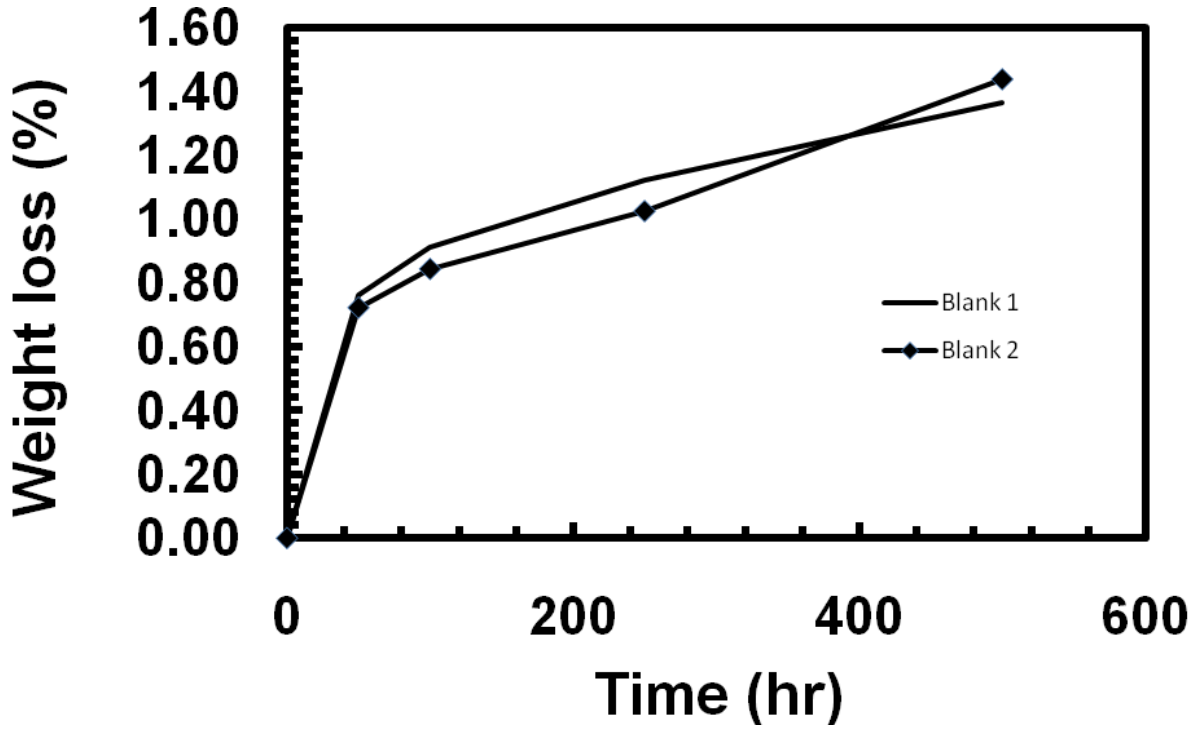


**Figure 27 Percent weight loss of rectangular specimens as a function of aging time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C**

A visual inspection of the specimens revealed no discoloration or evidence of degradation. A cross sectional cut was taken from each of the rectangular specimens using a diamond saw. The cut surface was polished and viewed under an optical microscope to determine if the specimens had undergone micro-structural changes. These specimens confirmed that the argon testing had performed within expectations. Oxidation at high temperatures appeared to be eliminated. Micrographs of the all specimens are presented in Appendix A.

*Elastic Modulus*

**Room Temperature Modulus**

Determination of the sample-to-sample variability required that the initial room temperature elastic modulus of each sample be measured and compared. This test was completed after machining but prior to any aging or elevated temperature testing. Individual specimens were loaded at a 45° orientation to 3 MPa at a rate of 1 MPa/s and unloaded to zero stress at the same rate. The covariance of the elastic modulus for all of the 24 specimens was determined to be .0817. This measurement was determined by dividing the standard deviation of 1.282 by the average elastic modulus of 15.684 GPa.

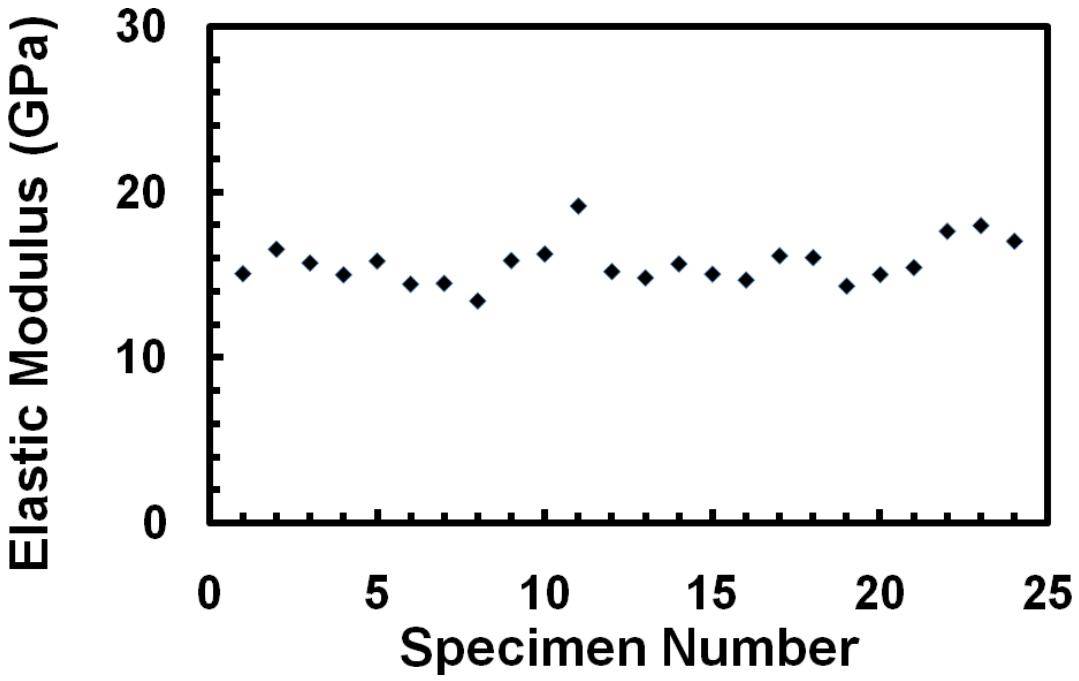


**Figure 28 Initial room temperature elastic modulus data as a function of specimen number of carbon fiber reinforced PMR-15 composite**

The scatter between the points was determined to be small enough so that no samples needed to be eliminated based solely on this measurement. Figure 28 shows the initial room temperature elastic modulus as a function of specimen numbers.

**Aged Modulus versus Initial Modulus**

The data necessary to calculate the elastic modulus at elevated temperature was obtained in order to evaluate the effects that aging has on the mechanical properties of carbon fiber PMR-15 composite. The same load rate of 1 MPa/s was used during the creep phase of the testing.

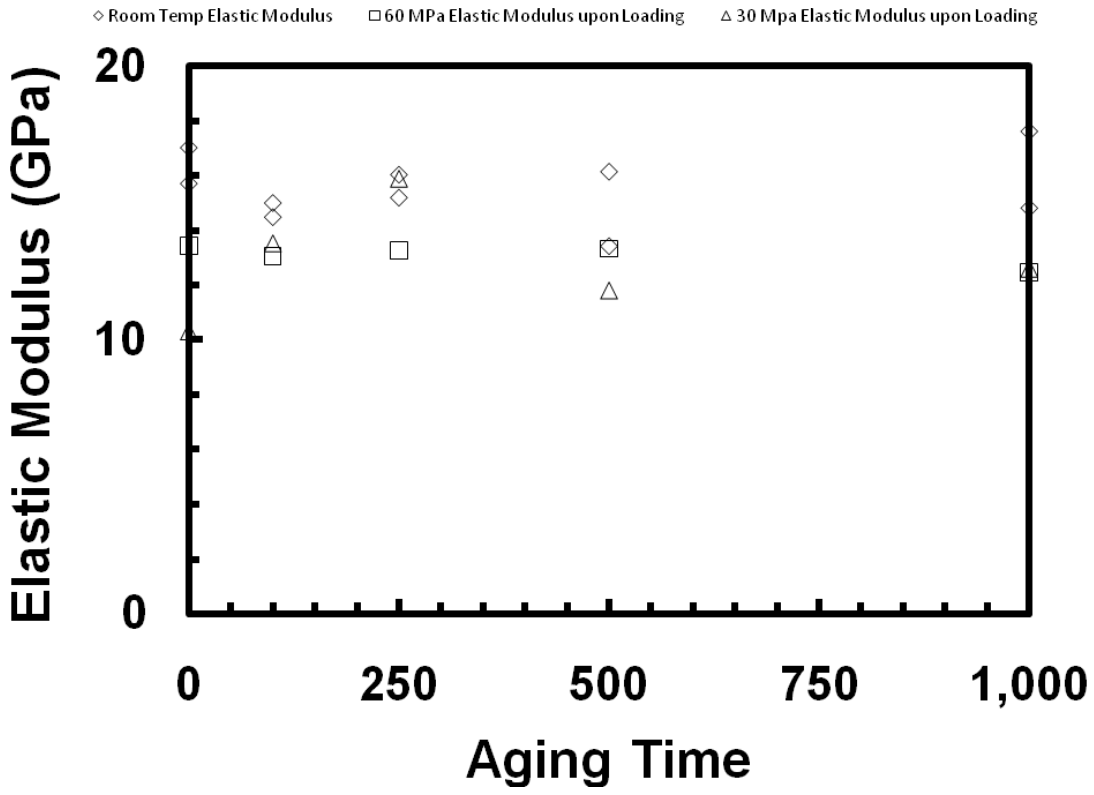


**Figure 29 Elastic modulus of aged and unaged room temperature samples as a function of aging time for carbon fiber reinforced PMR-15 composite**

The elastic modulus for each specimen was calculated from the stress curve between 0 and 7 MPa. The room temperature modulus was higher than the aged 30 MPa and 60 MPa loaded modulus. Figure 29 shows the elastic modulus at room temperature loading, aged 30 MPa loading, and aged 60 MPa loading measured at 288°C.

**Elastic Modulus upon Loading and Unloading**

The stress-strain curves for loading and unloading were determined for all specimens. It is evident from Figure 30 that the aged unloading elastic modulus is generally lower than the aged loading elastic modulus. This can be accredited to the damage accumulated during mechanical testing.

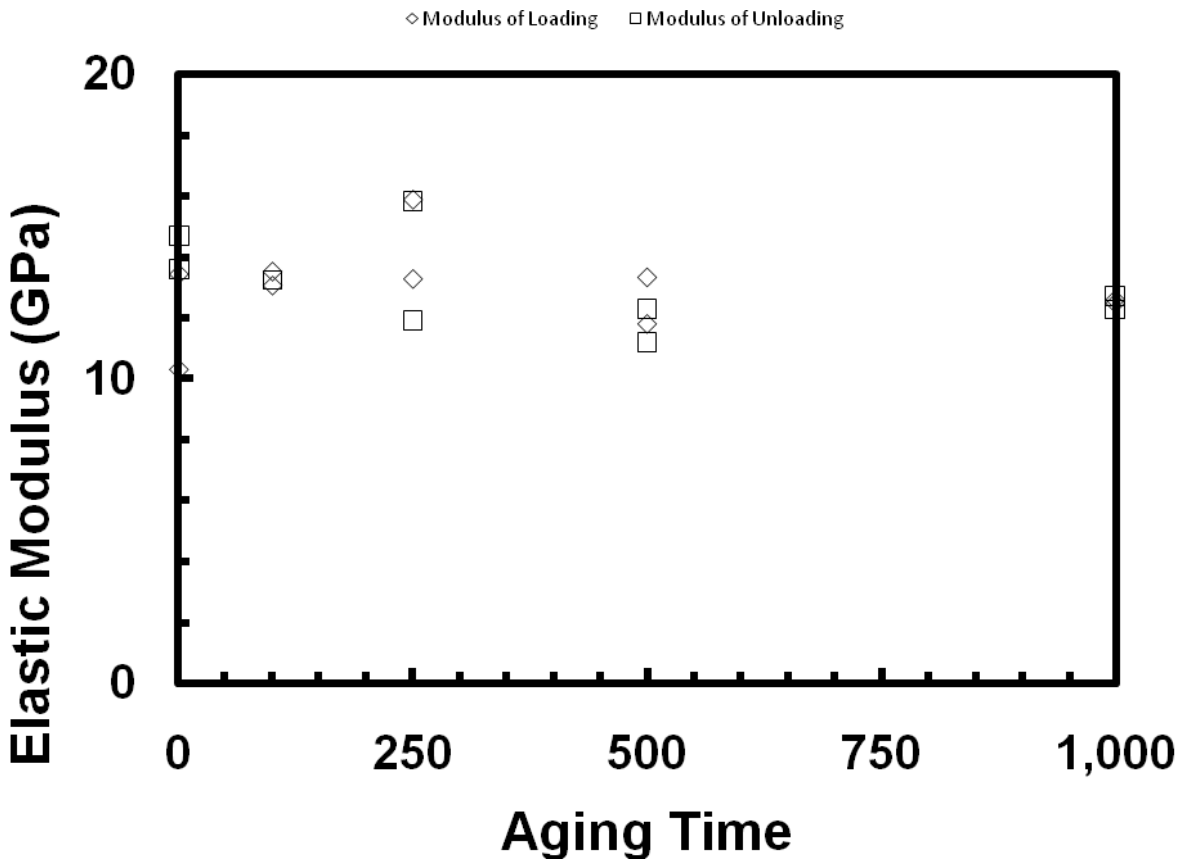


**Figure 30 Modulus of loading and Modulus of unloading as a function of aging time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C**

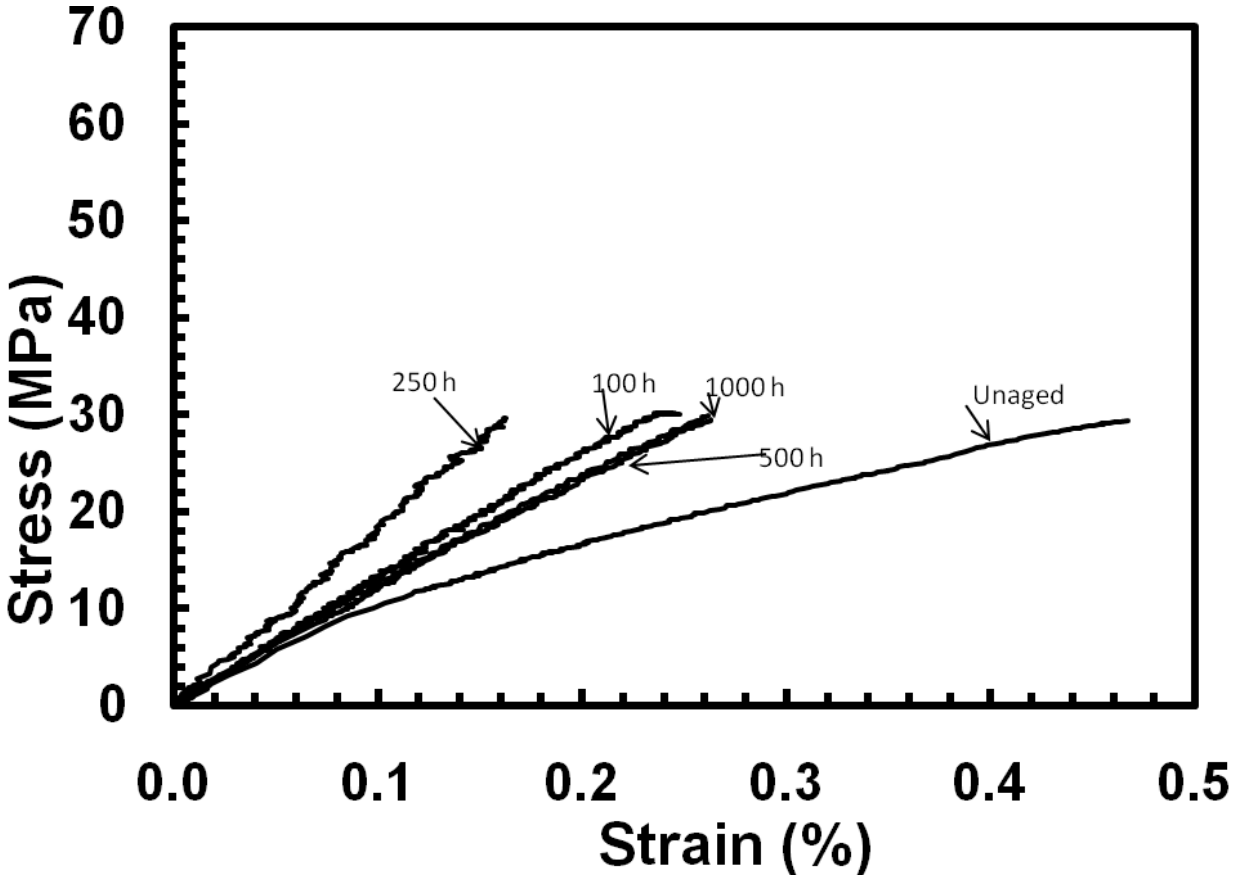


**Figure 31 Tensile stress-strain curves during loading at a 45° orientation of carbon fiber reinforced PMR-15 composite aged in argon at 288°C to a stress of 30 MPa**

The tensile stress-strain curves for a load of 30 MPa and a load of 60 MPa can be seen in Figures 31 and 32 respectively. It appears that there is a general increase in the elastic modulus from 100 to 500 hours. This suggests that the bulk of the material is stiffening. Previous research suggests that this trend may continue through 1000 hours [6], although this data shows the elastic modulus decreasing from 500 to 1000 hours.

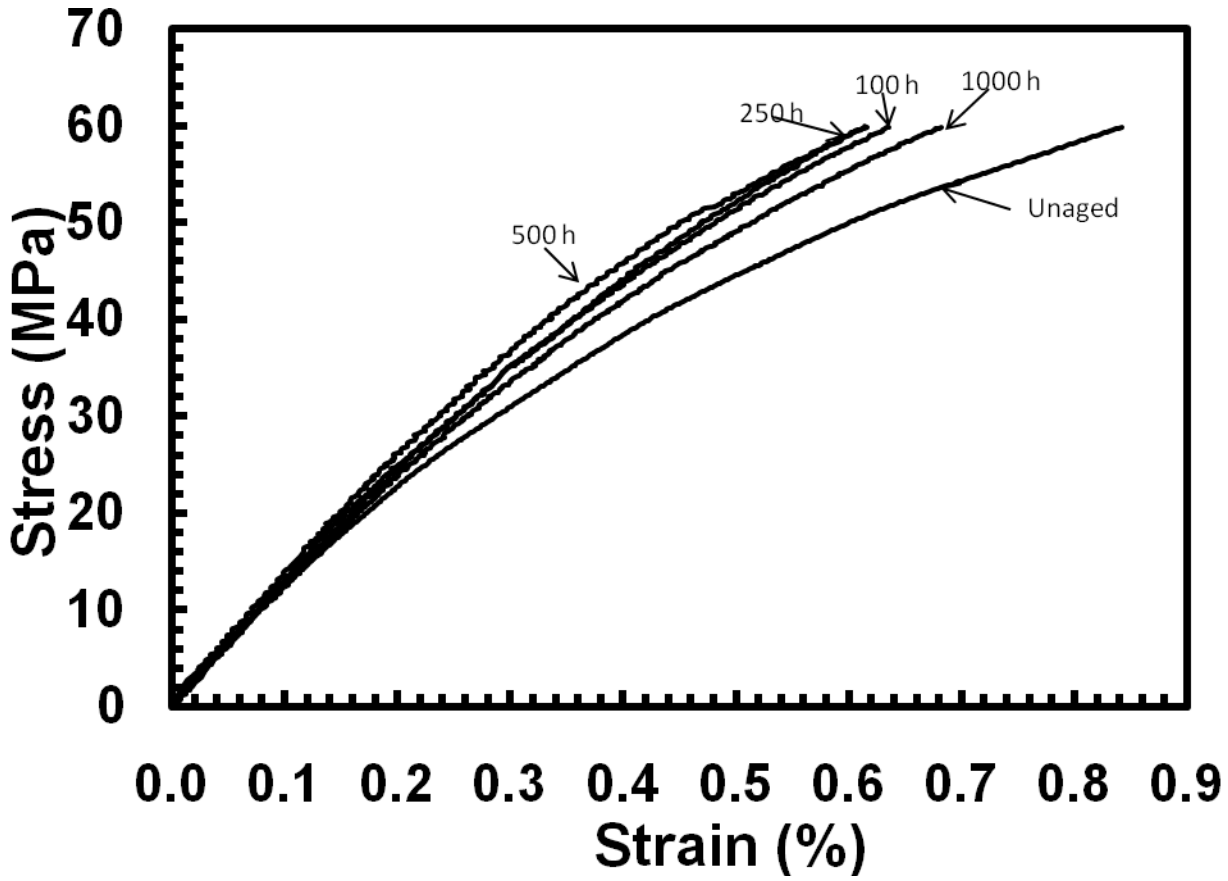


**Figure 32 Tensile stress-strain curves during loading of carbon fiber reinforced PMR-15 composite aged in argon at 288°C to a stress of 60 MPa**

Figure 33 shows the elastic modulus measurements during unloading as a function of aging time in argon at 288°C. The results have been broken down by creep stress, as it is possible for the unloading modulus to be influenced by prior creep. The elastic modulus does not appear to show any particular trend. Were the modulus values to decrease, it could be concluded that the creep period was causing structural damage to the specimens.

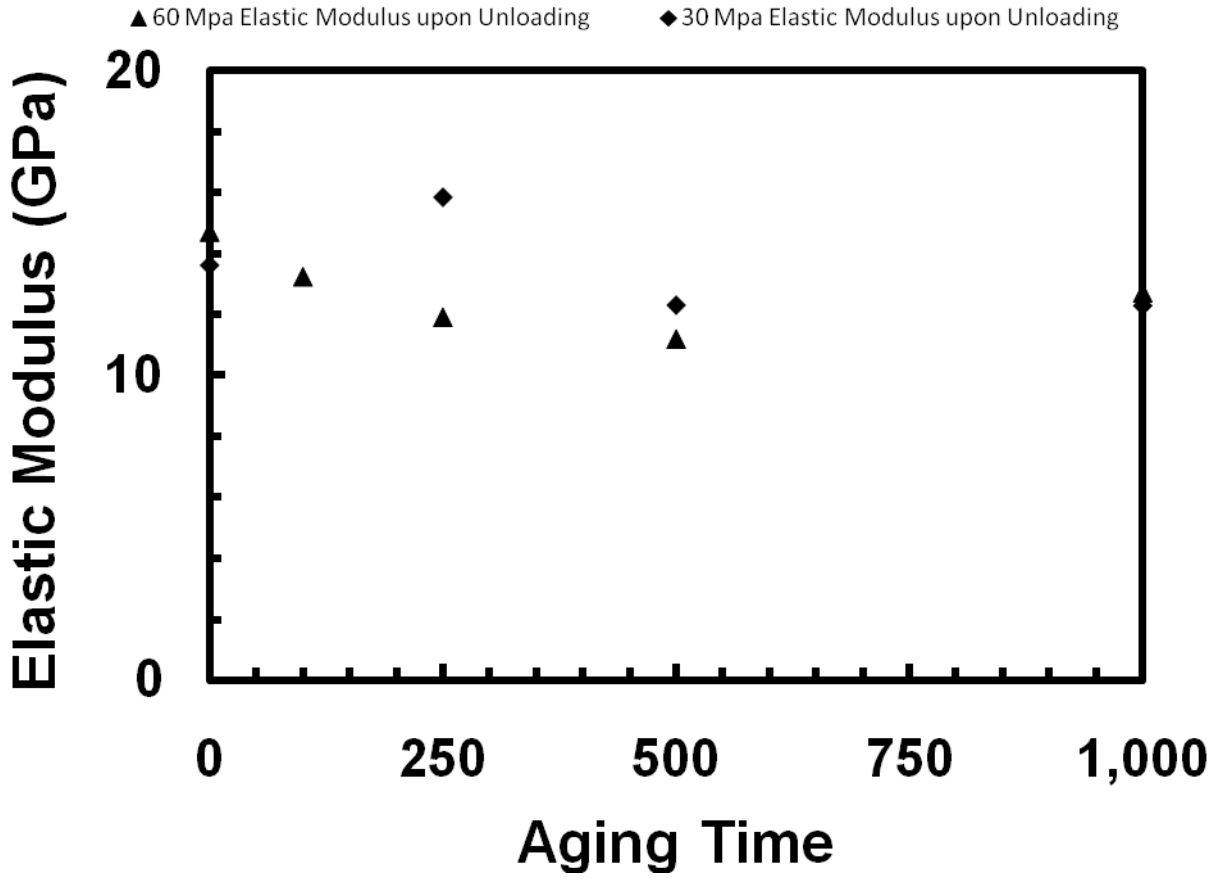


**Figure 33 Elastic modulus measurements upon unloading as function of aging time of carbon fiber reinforced PMR-15 composite aged in argon at 288°C**

The % change in modulus between loading and unloading as a function of aging time is shown in Figure 34. The purpose of this calculation is to understand the effect that the specimen's modulus of loading has on its modulus of unloading. The maximum specimen variation was 25% in the modulus of loading and the modulus of unloading.

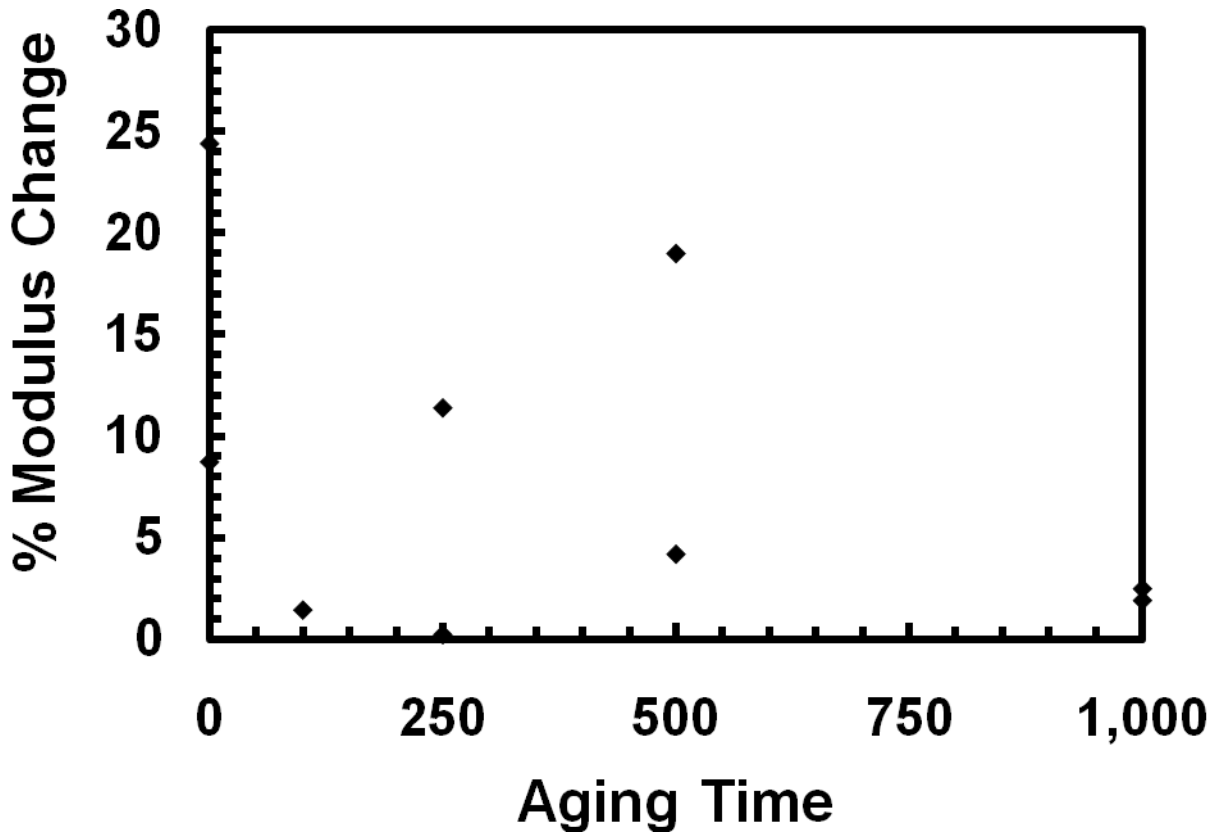


**Figure 34 Change in elastic modulus between loading and unloading as a function of aging time of carbon fiber reinforced PMR-15 composite aged in argon at 288°C**

**Elastic Modulus Ratios**

The dynamic material changes due to aging and mechanical loading are of particular interest to this research. A necessary step in order to identify those properties is to measure the ratios of elastic moduli before, during, and after testing. Different formulations of these moduli result in a large quantity of useful data. Examination of the ratio of the room temperature elastic modulus to the elastic modulus during loading will result in information on the effects of increased temperature and aging time on the elastic response. Indication of damage developed in the material during the creep period can be

found with a ratio of the modulus during unloading to the modulus during loading. To determine the material changes induced by the entire aging and testing process, the ratio of the modulus during unloading to the initial modulus can be inspected. Figure 35 shows these results for a stress of 60 MPa as a function of aging time.

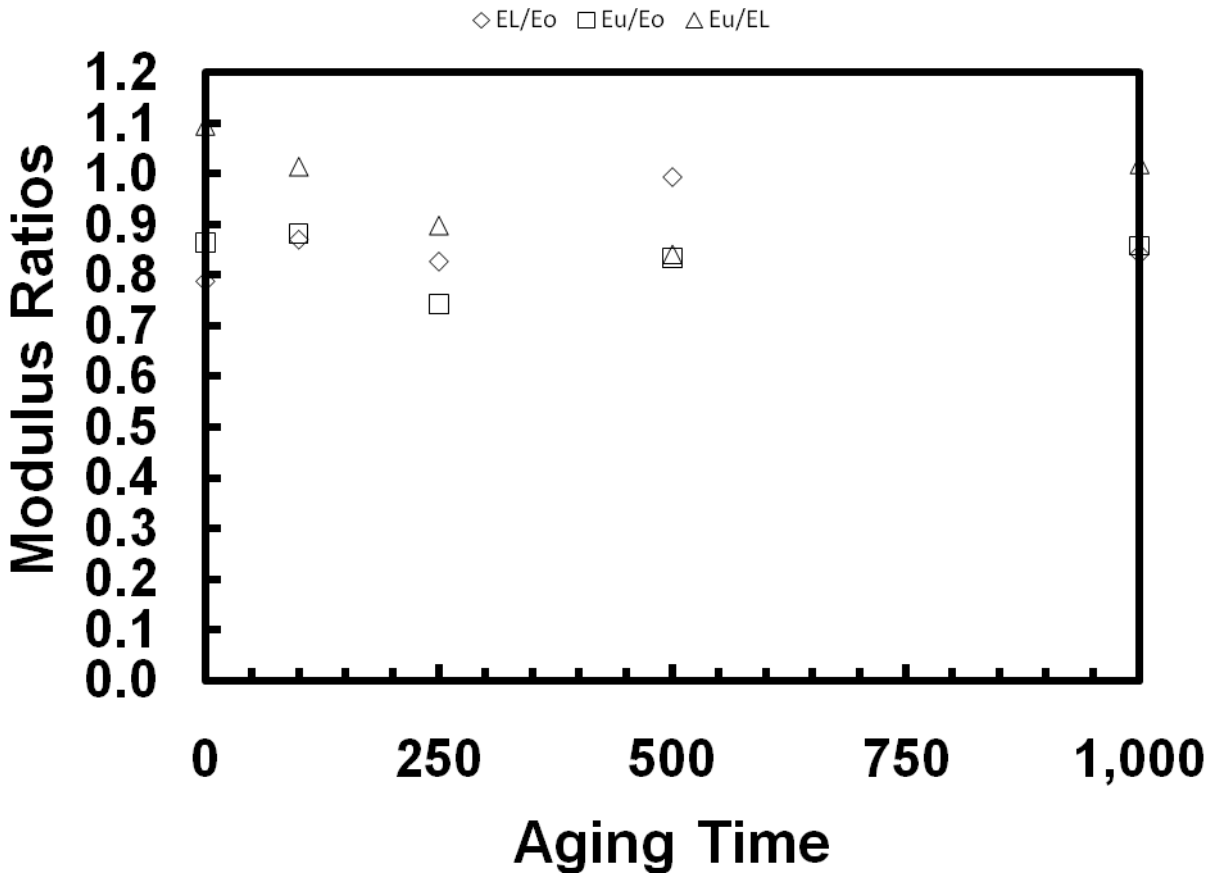


**Figure 35 Elastic modulus ratios versus aging time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C and tested in creep at 60 MPa**

Figure 35 shows very little variation of the modulus ratios with aging time.

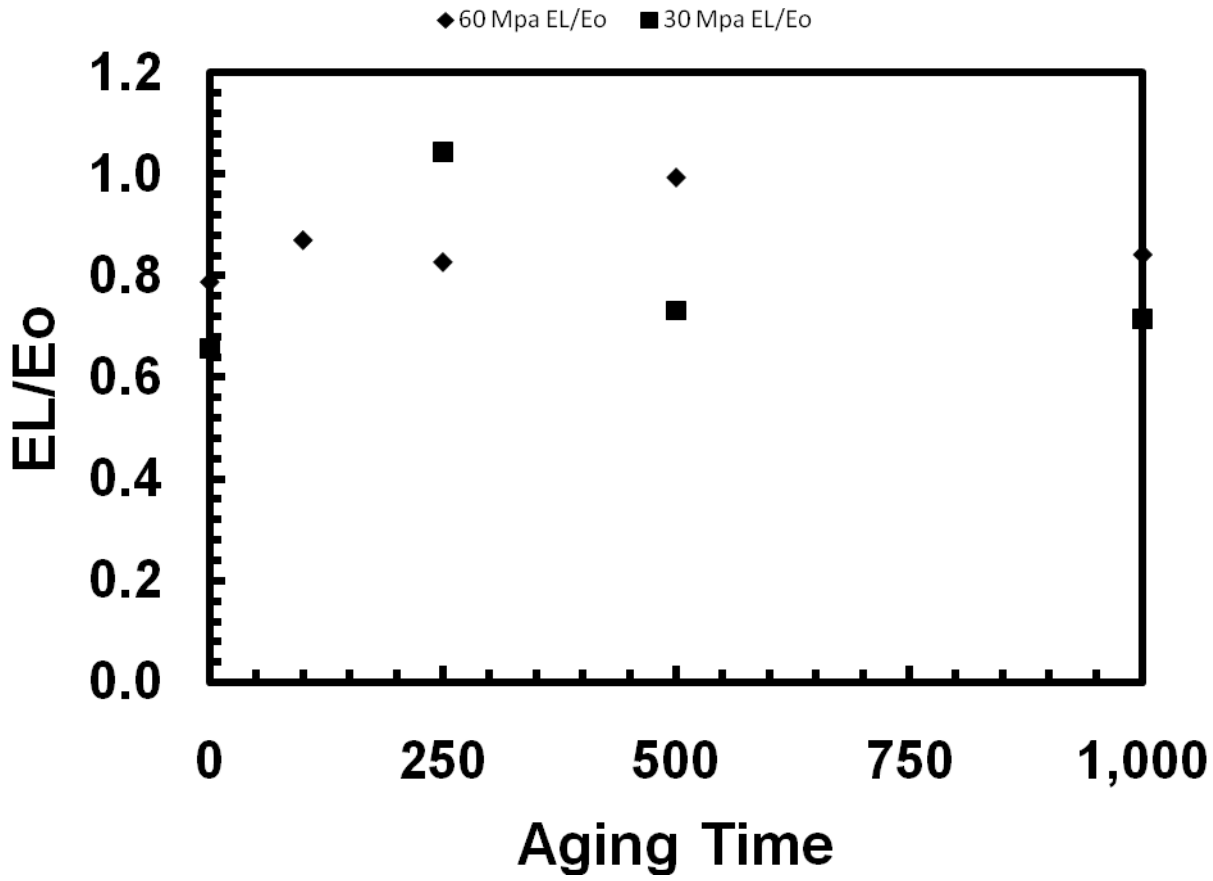


**Figure 36 Ratio of elastic modulus measured upon loading to the initial room temperature elastic modulus as a function of aging time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C and tested in creep at 30 and 60 MPa**

The ratio of the elastic modulus upon loading to the initial elastic modulus as a function of aging time is shown in Figure 36. This represents the effect that temperature increase as well as aging has on the specimens. The 30 MPa loaded specimen increases from 0 hours to 250 hours, and then decreases to 500 hours until finally leveling at approximately .7 at 1000 hours. The data point at 250 hours is surprising and most likely represents abnormal specimen. The ratio of the 60 MPa loaded specimen rises from 0 hours to 100 hours and then decreases to 250 hours rises to 500 hours and decreases again

to 1000 hours. The relatively large fluctuation in values suggests that the aging time and temperature do in fact have a significant effect on the elastic properties of the specimens.

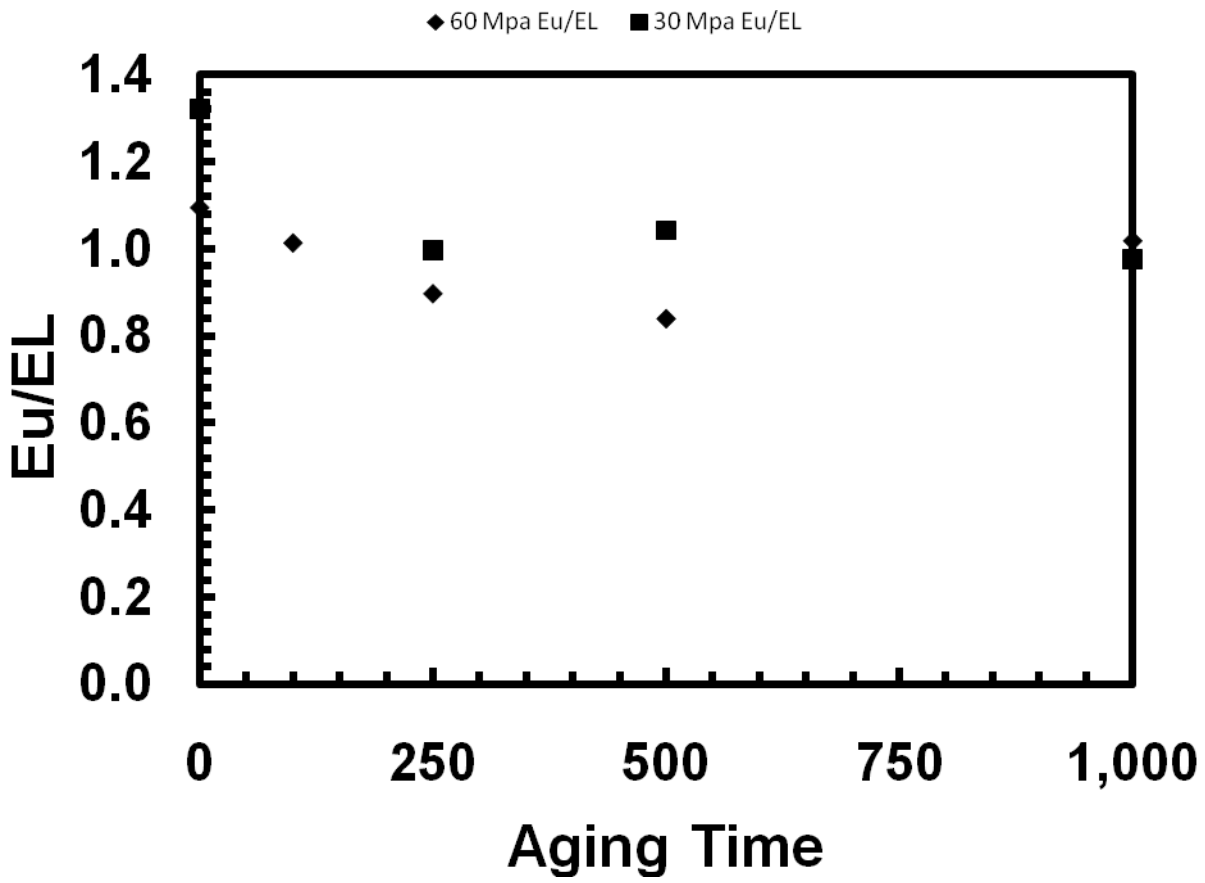


**Figure 37 Ratio of elastic modulus measured upon unloading to the elastic modulus measured upon loading as a function of aging time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C tested in creep at 30 and 60 MPa**

Figure 37 results in an observation that the ratio of the elastic modulus measured upon unloading to the elastic modulus upon loading is not dependent on the creep stress level. This is intriguing because previous research [6] showed dependency on creep stress for PMR-15 without carbon fiber reinforcement. Damage is apparent in both loading

groups, although not in every specimen. Specimens tested at 60 MPa appear to have a higher reduction in elastic modulus than similarly aged specimens at a load of 30 MPa.

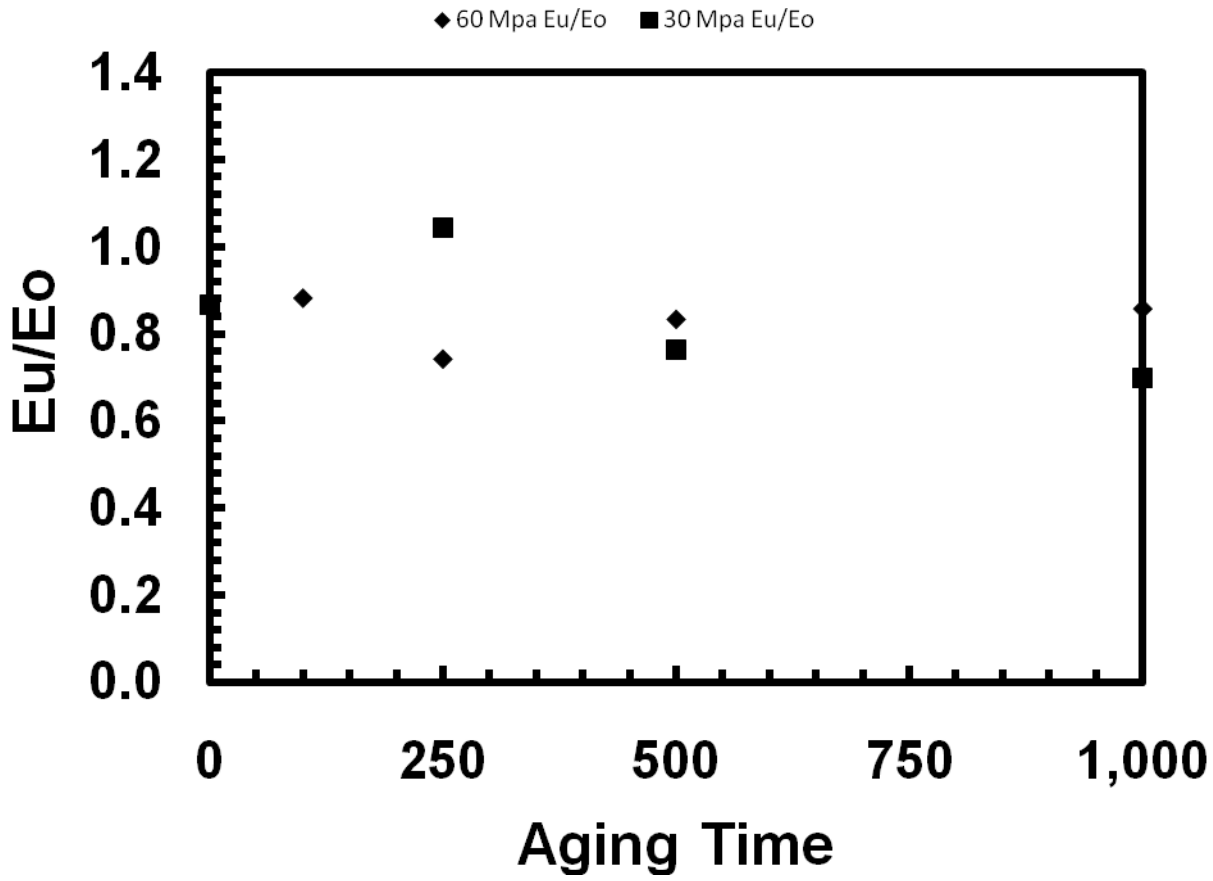


**Figure 38 Ratio of elastic modulus measured upon unloading to the initial room temperature elastic modulus as a function of aging time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C tested in creep at 30 and 60 MPa**

The ratio of the modulus during unloading to the initial modulus as a function of aging time is shown in Figure 38. This ratio shows the largest amount of damage to the elastic modulus as it is the damage accrued from prior aging, creep loading, and elevated temperature.

These ratios may be a more accurate representation of the degree of degradation than the actual plotted modulus values. They allow mechanical damages due to thermal degradation, aging, and loading history to be separated and evaluated in a way that is not possible from examination of the modulus changes alone. This method of evaluation grants a greater degree of understanding of the damages incurred by the testing process.

### *Monotonic Tensile Tests*

#### Tensile Tests for Aged and Unaged Specimens

Tensile tests to failure were carried out on the carbon fiber reinforced PMR-15 composite specimens for each age group in order to ascertain the ultimate tensile strength of the material. Prior research noted that the ultimate tensile strength may not be reached due to fractures caused by the dimples in each specimen [1]. The purpose of this procedure was to find Young's Modulus of Elasticity and the Ultimate Tensile Strength. Figure 39 shows the stress-strain curves of all tested specimens.

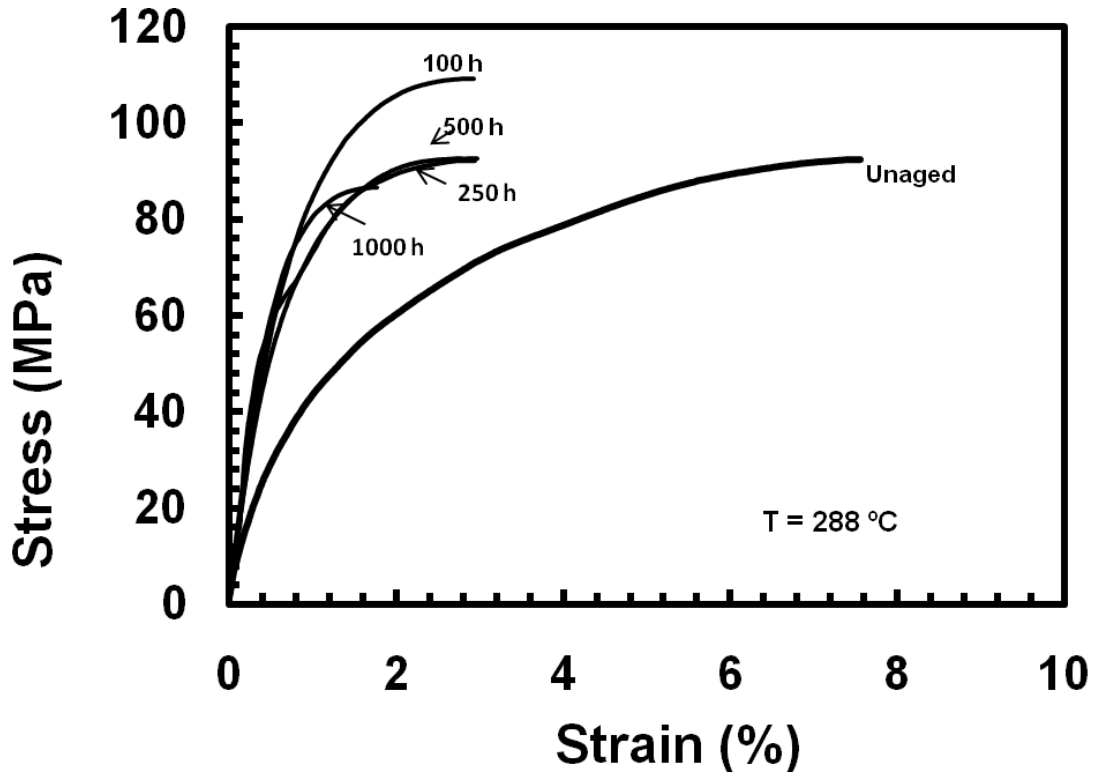


**Figure 39 Stress-strain curves for aged and unaged specimens at 288°C during tensile testing**

| Aging | Modulus (GPa) | Ultimate Tensile Strength |
|---|---|---|
| Unaged | 15.429 | 92.398 |
| 100 h | 14.029 | 109.124 |
| 250 h | 13.097 | 92.044 |
| 500 h | 12.938 | 92.662 |
| 1000 h | 16.058 | 86.451 |

**Table 4 Elastic modulus and UTS measurements for test specimens subjected to tensile testing at 288°C**

Table 4 shows the difference in the modulus of loading between the specimens. These modulus values were calculated from the portion of the stress-strain curves from 0 to 10 MPa for each specimen. The table also indicates the trend of the Ultimate Tensile Strength to decrease in value as the aging time increases. It can be seen that the UTS actually increased from the unaged specimen to the 100 hour specimen. This is a

surprising result as the modulus of each was very similar. Every specimen failed in the gage section, immediately preceded by fiber scissoring. This scissoring accounted for large failure strain. Figure 40 shows the strain as a function of time that it took for each specimen to fracture.

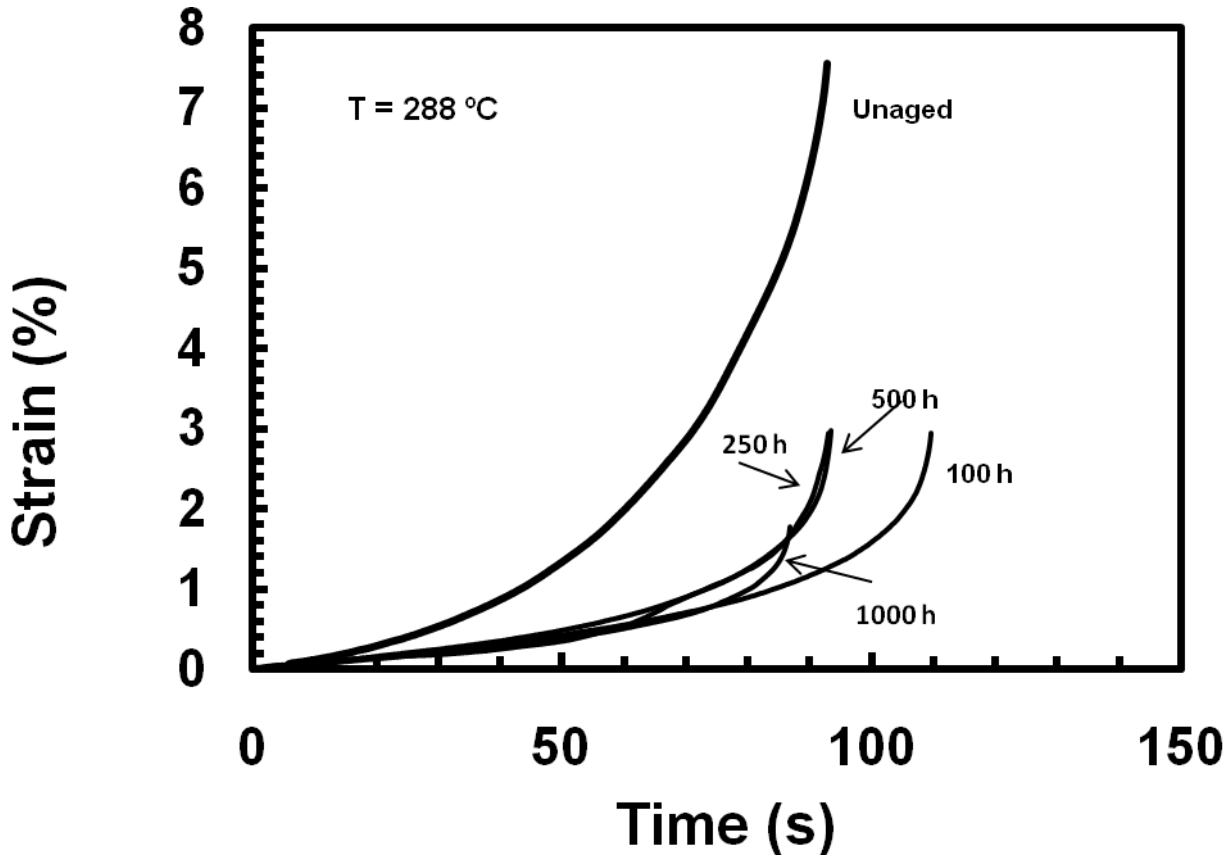


**Figure 40 Strain to fracture as a function of time for carbon fiber reinforced PMR-15 composite specimens at 288°C**

## Creep Tests of Unaged Specimens

Creep studies were conducted at stress levels of 30 MPa and 60 MPa for a period of 25 hours followed by unloading to zero stress for a recovery period of 50 hours. The results of these tests are plotted as creep strain as a function of creep time. The stress was forced to remain constant during the creep tests, but the strain increased. Figure 41 shows the creep curves of the unaged specimens loaded to 30 MPa and 60 MPa as a function of time at 288°C.

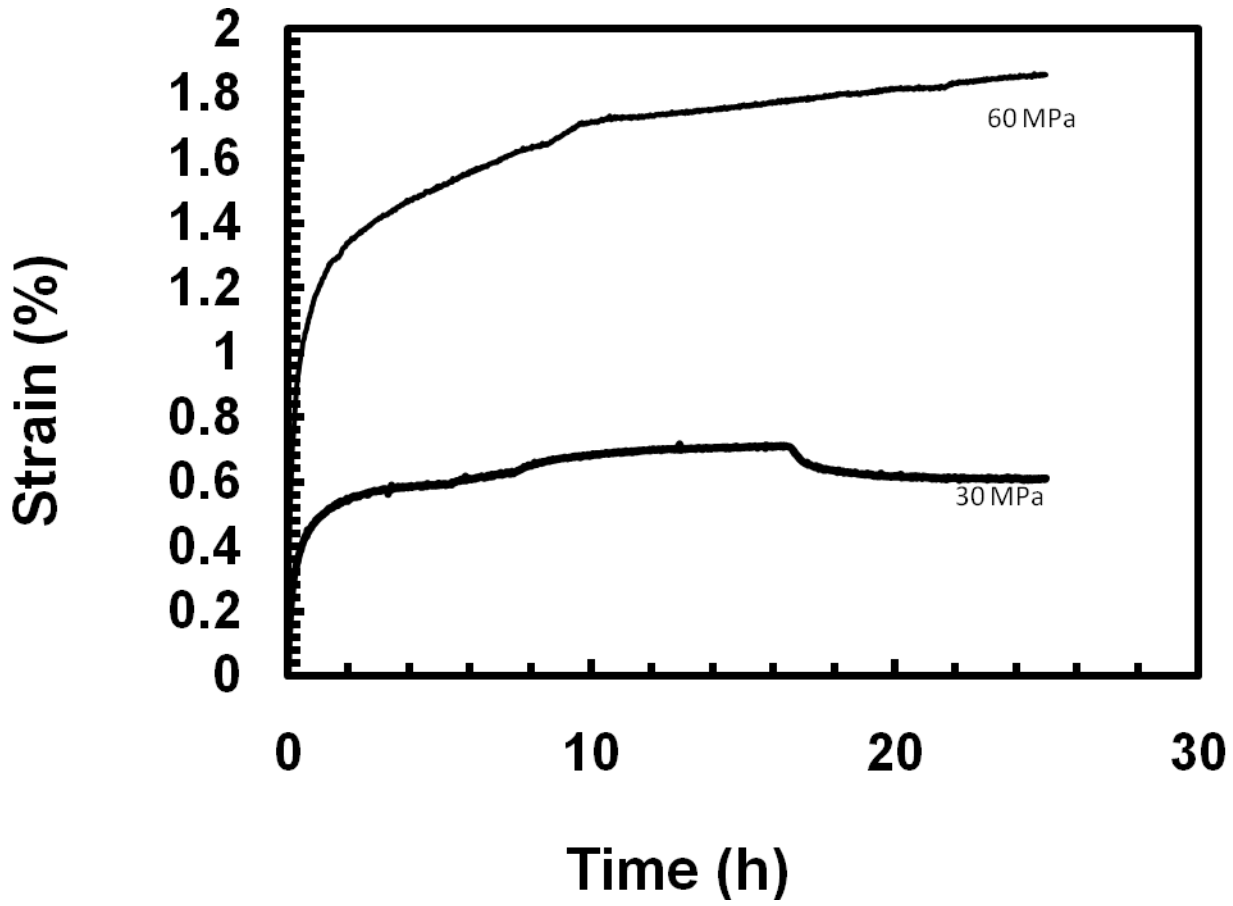


**Figure 41 Creep strain as a function of time for unaged carbon fiber reinforced PMR-15 composite for a creep stress level of 30 MPa and 60 MPa at 288°C**

It can be seen in Figure 41 that higher creep stress levels caused higher creep strains to be produced in the specimens. Primary and secondary creep regimes are present. The transition period for both specimens appears to be approximately 3 to 4 hours. Note that the secondary creep rate or steady-state rate increased with an increase in creep stress level.

**Creep Tests at 60 MPa**

Creep and recovery tests were conducted on specimens aged for varying periods of time in an argon environment at 288°C. A creep stress level of 60 MPa was maintained for a period of 25 hours, immediately followed by a recovery period at zero stress of 50 hours. Figure 42 shows the results of the individual tests in the format of creep strain as a function of creep time. Past research has shown that the creep strain accumulation decreases with increased aging time [6, 7]. This trend is apparent in the figure, except for the 100 hour aged specimen. The creep strain of ~.8% that this specimen accumulated is not in line with the established response. This is a curious response as the initial room temperature elastic modulus of this specimen was close to the average value for this specimen set. This anomaly can perhaps be accredited to a defect inherent to that particular specimen. The unaged specimen accumulated the most creep strain at ~1.9%, followed by the 250 hour specimen at ~1.2%, the 500 hour specimen at ~1%, and the 1000 hour specimen at ~.7%. This data provides verification of the assertion that increased aging time decreases the amount of creep strain accumulation.

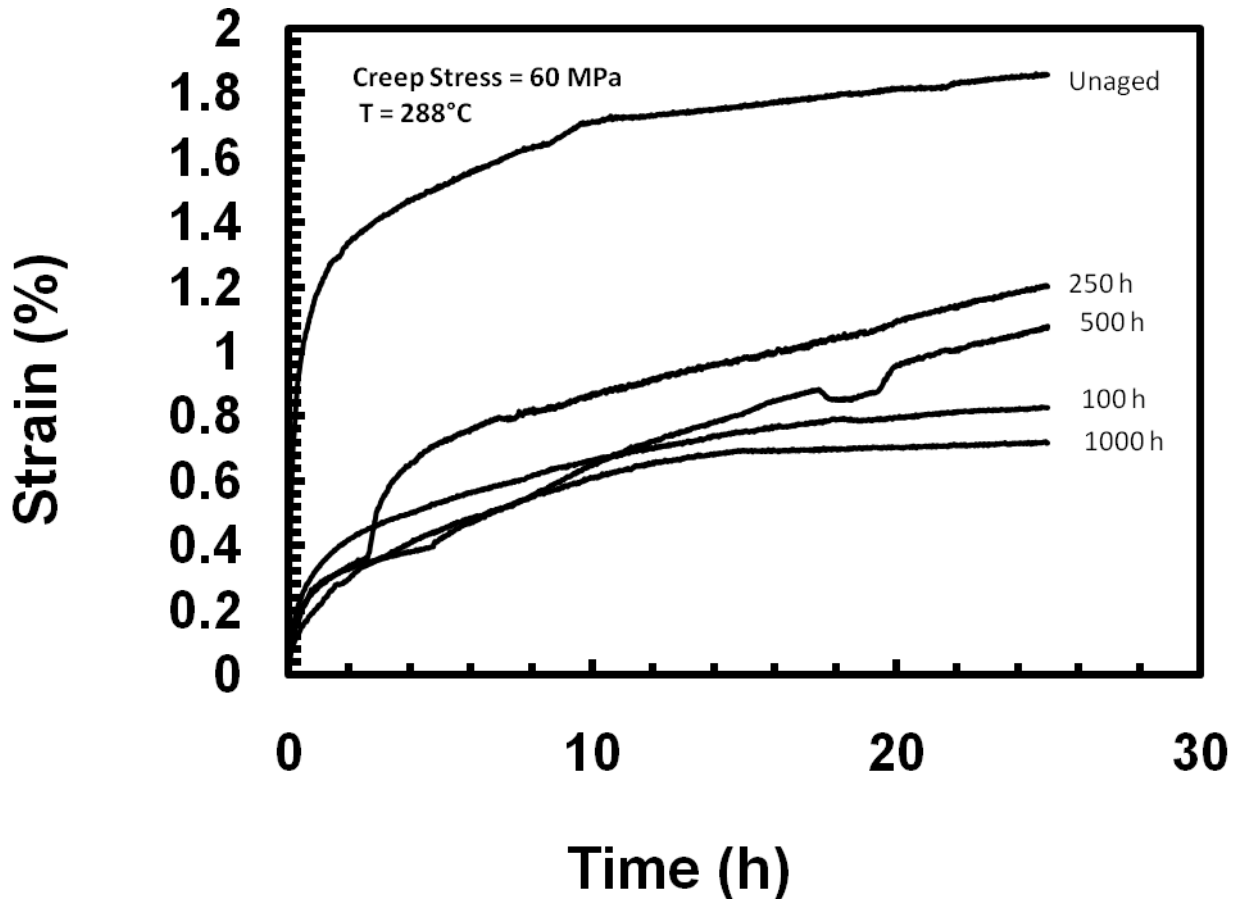


**Figure 42 Creep strain as a function of time for carbon fiber reinforced PMR-15 composite tested in creep at 60 MPa at 288°C**

**Creep Tests at 30 MPa**

Creep and recovery tests were conducted on specimens aged for varying periods of time in an argon environment at 288°C. A creep stress level of 30 MPa was maintained for a period of 25 hours, immediately followed by a recovery period at zero stress of 50 hours. The results of these tests are shown in Figure 43 in the form of creep strain as a function of creep time. As can be seen in the Figure, influence of aging time is less pronounced than the 60 MPa creep tests. It is worth noting that the 100 hour specimen accumulated a creep strain of ~.06, a value well below what might be expected from the established response.

The 500 hour specimen is also slightly above the expected value range or alternatively the 250 hour specimen is slightly below its expected value. This seems to indicate that one of these specimens contained an unknown defect as well. The other specimens appear to follow the accepted norm for tests of this nature in that it is clear to see the trend of increased aging time causing a decrease in the amount of creep strain accumulation. The spikes in the graphs emerge from the low stress level being below the tolerance of the noise inherent to the system.

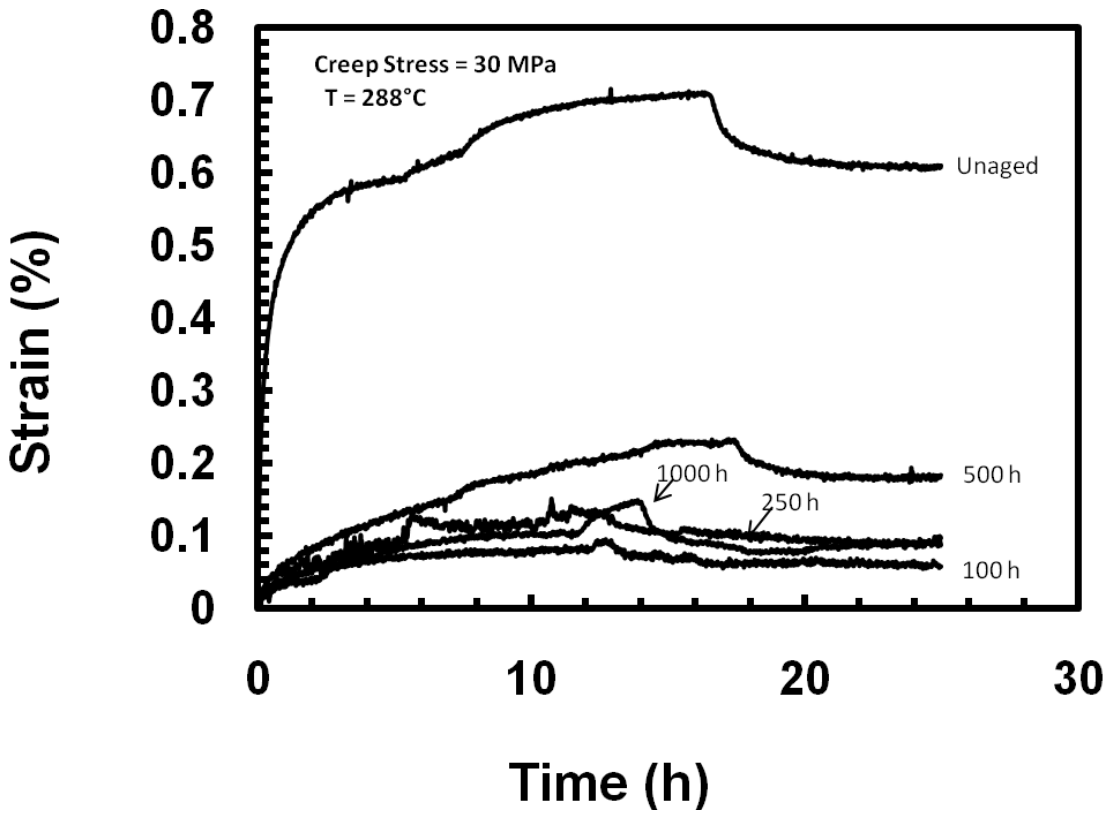


**Figure 43 Creep strain as a function of time for carbon fiber reinforced PMR-15 composite for a creep stress level of 30 MPa at 288°C**

Figure 44 plots the strain accumulated in each test after 25 hours of creep as a function of the aging time for both creep stress levels of 30 and 60 MPa, respectively. This is a useful plot that allows for a closer examination of the argument that increased aging time decreases the strain accumulation.

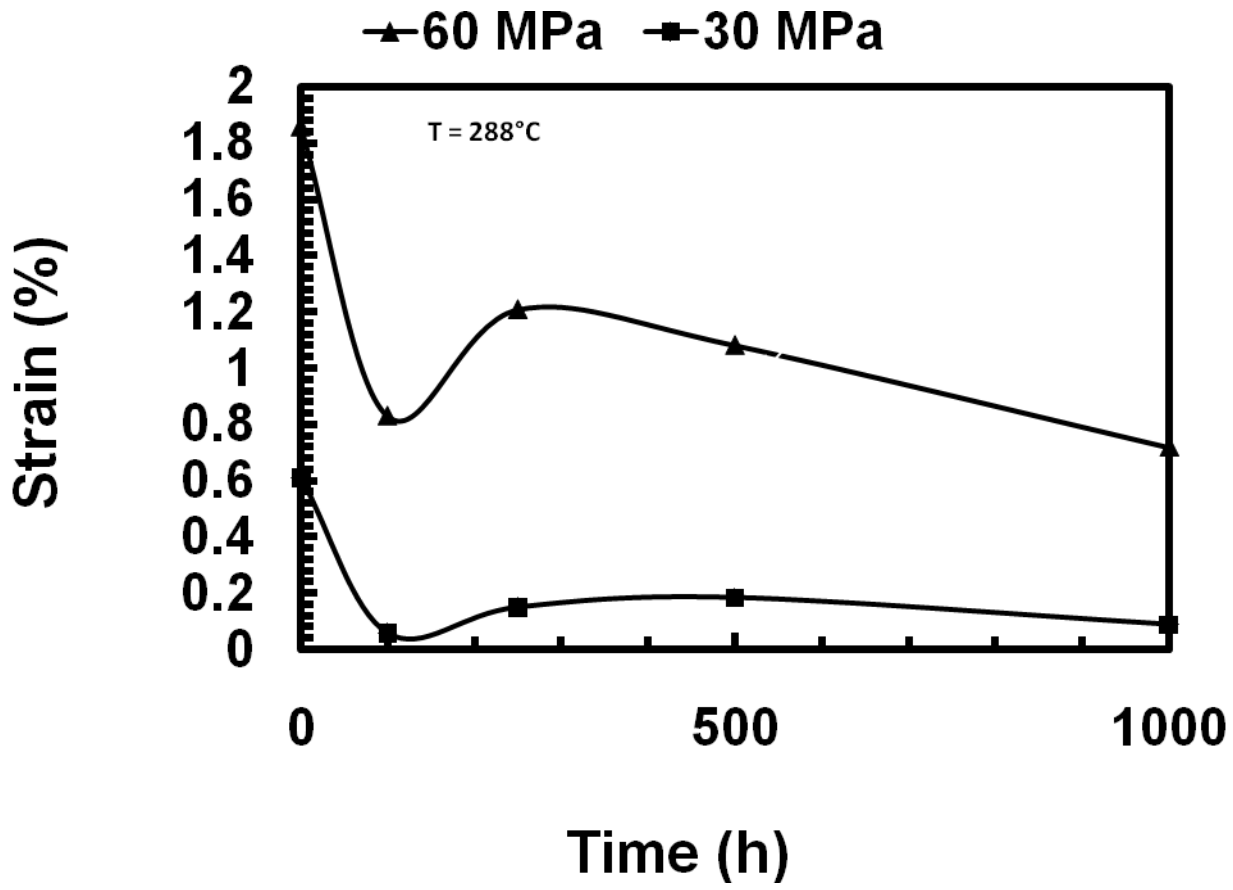


**Figure 44 Creep strain accumulated in 25 hours as a function of aging time for carbon fiber reinforced PMR-15 composite at 288°C**

It can be seen that the 1000 hour specimen accumulated 50% less creep strain than the unaged specimen. The 100 hour specimens for both stresses appear to be following a similar trend. This could be attributed to a post-cure like response in the 100 hour specimens where the material properties change for the first 100 hours of aging and then

begin the degradation process. The general trend to the data that was observed in the 30 MPa and 60 MPa creep tests is clear. There is an inverse relationship between accumulated creep strain and prior aging time.

**Recovery at Zero Stress for 60 MPa**

Creep and recovery tests were conducted on specimens aged for varying periods of time in an argon environment at 288°C. A creep stress level of 60 MPa was maintained for a period of 25 hours, immediately followed by a recovery period at zero stress of 50 hours. Examination of Figure 45 shows the amount of strain recovered as a function of recovery time. It is easy to see that there is an initial period of rapid strain recovery, followed by a period where the recovery rate slowly decreases to a constant value. This indicates that the recovery process achieved saturation during the 50 hour recovery period. The amount of chemical, physical and thermal damages accumulated during the testing of this material can be inferred from this figure. The unaged specimen recovers the most as it had the least amount of damage. The amount of strain recovery appears to decrease as the amount of prior aging increases. The 100 hour specimen is still showing unexpected results, in that it recovers less than the 250 hour specimen and is in fact on par with the 500 hour specimen strain recovery amount.

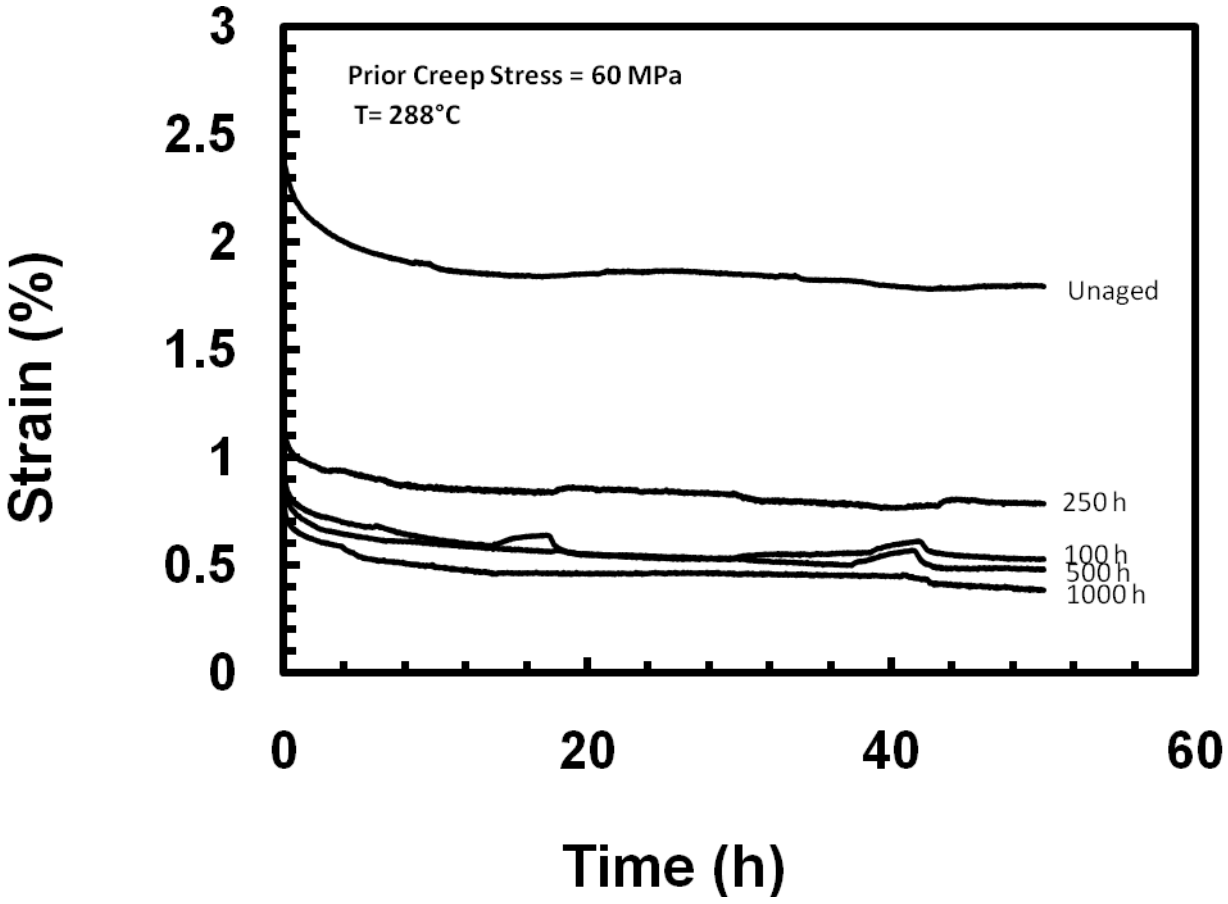


**Figure 45 Recovery curves for carbon fiber reinforced PMR-15 composite aged in argon at 288°C with a prior creep stress of 60 MPa at 288°C**

Comparison of the recovery curves in Figure 45 is difficult as the placement on the plot is dependent upon the amount of strain accumulated during the creep testing period. However, as it is important to examine the amount of recovered creep, another method of examination is necessary. A schematic depiction of the stress-strain curve for a constant stress creep period is shown in Figure 46. Recovered strain is defined as $\varepsilon^{r} = \varepsilon^{'} - \varepsilon^{*}$, where $\varepsilon^{'}$ and $\varepsilon^{*}$ are the strain after the start of the recovery period and at some time t during the recovery period, respectively.

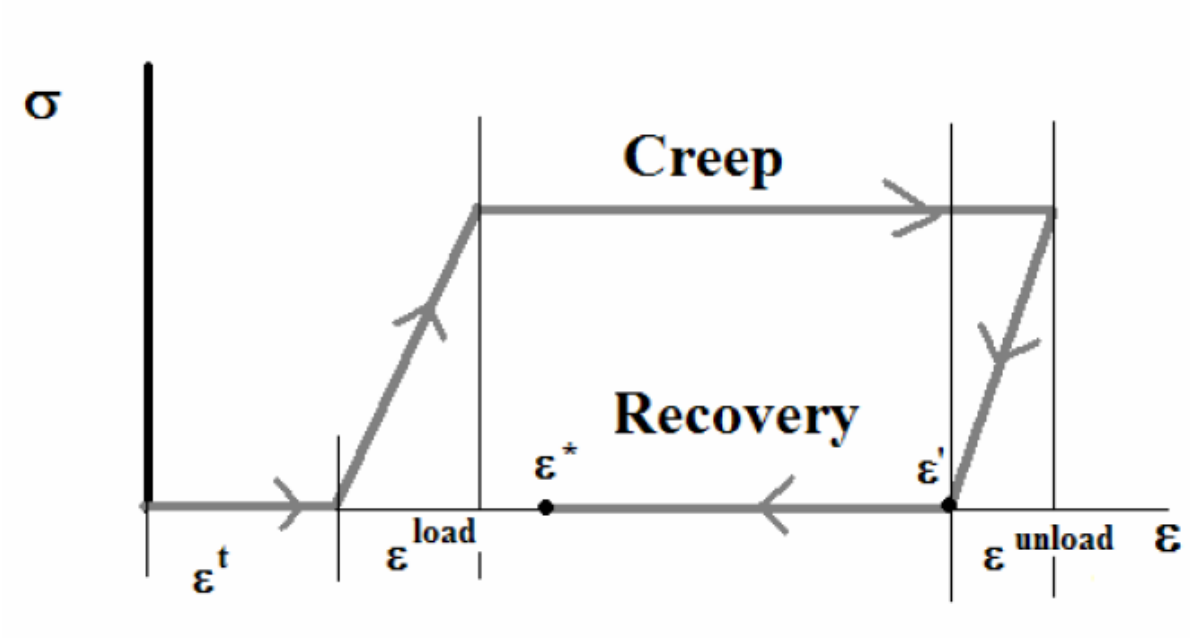


**Figure 46 Schematic stress-strain curve for constant stress creep period followed by recovery period at zero stress. Figure from Ref. [6]**

The recovered strain as a function of time is shown in Figure 47 for all specimens tested at a 60 MPa creep stress level. The recovered strain for the specimens at a creep stress level of 30 MPa is not shown graphically because the data recovered is approaching the accuracy of the extensometer. The data is overwhelmed by the electrical noise inherent to the system and is misleading.

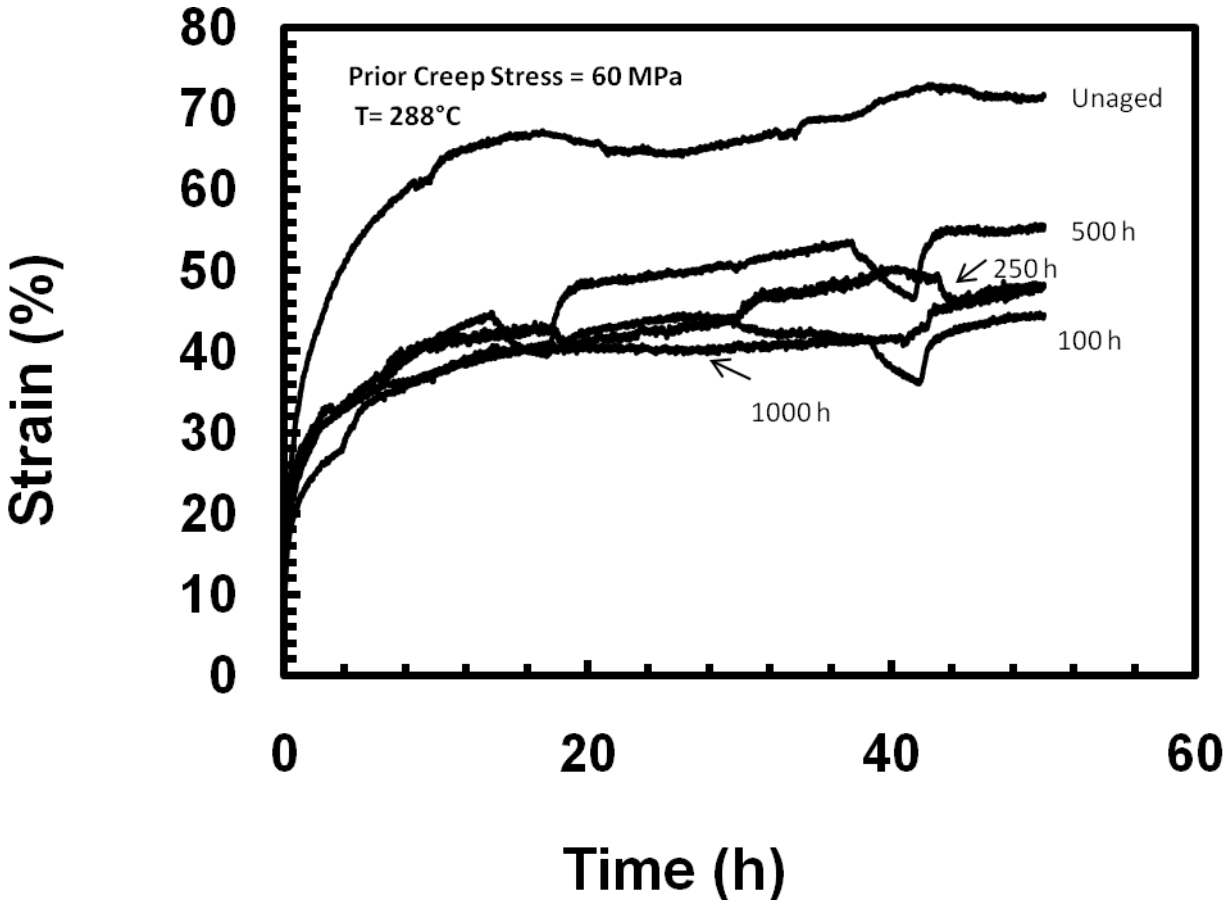


**Figure 47 Recovered strain as a function of time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C and loaded to a creep stress of 60 MPa at 288°C**

Figure 47 clearly shows that the unaged specimen is recovering much more of the strain accumulated during creep than any of the other specimens. This matches data gathered via previous research [6]. An inverse relationship exists between the recovered strain and the amount of prior aging. The other specimens recovered approximately 40 to 50% of their accumulated strain.

This apparent trend could be explained as a function of the amount of strain to be recovered. The amount of creep strain accumulated by specimens that were unaged or aged for a short period of time could have simply been higher than the longer aged

specimens. Therefore there would be more strain to recover, and the apparent relationship between the recovered strain to aging time would not exist in this data. In order to verify the results, it is useful to examine the recovered strain in relation to the amount of previously accumulated creep strains.

The percent of creep strain recovered was calculate using the schematic in Figure 46. The thermal strain accumulated during heating of the specimen to 288°C is designated as $\varepsilon^t$. Here, $\varepsilon^{load}$ is the strain accumulated during loading, $\varepsilon^{unload}$ is the strain accumulated during unloading, $\varepsilon^{'}$ is the strain value immediately upon reaching zero stress, and $\varepsilon^{*}$ is the instantaneous strain at any point in time during the recovery time. The strain accumulated during creep, $\varepsilon^{creep}$, is given by $\varepsilon^{creep} = \varepsilon^{'} - \varepsilon^{t} + \varepsilon^{unload} - \varepsilon^{load}$. Generally it is safe to assume that the value of $\varepsilon^{load}$ was equal to the value of $\varepsilon^{unload}$, so the equation for $\varepsilon^{creep}$ simplifies to $\varepsilon^{creep} = \varepsilon^{'} - \varepsilon^{t}$. The actual value of the percent of creep strain recovered is the ratio of recovered strain ($\varepsilon^{r} = \varepsilon^{'} - \varepsilon^{*}$) to creep strain ($\varepsilon^{creep} = \varepsilon^{'} - \varepsilon^{t}$). The results of these calculations can be found in Figure 48, which shows the percent of creep strain recovered as a function of recovery time for specimens aged in argon at 288°C and tested at a creep stress level of 60 MPa.

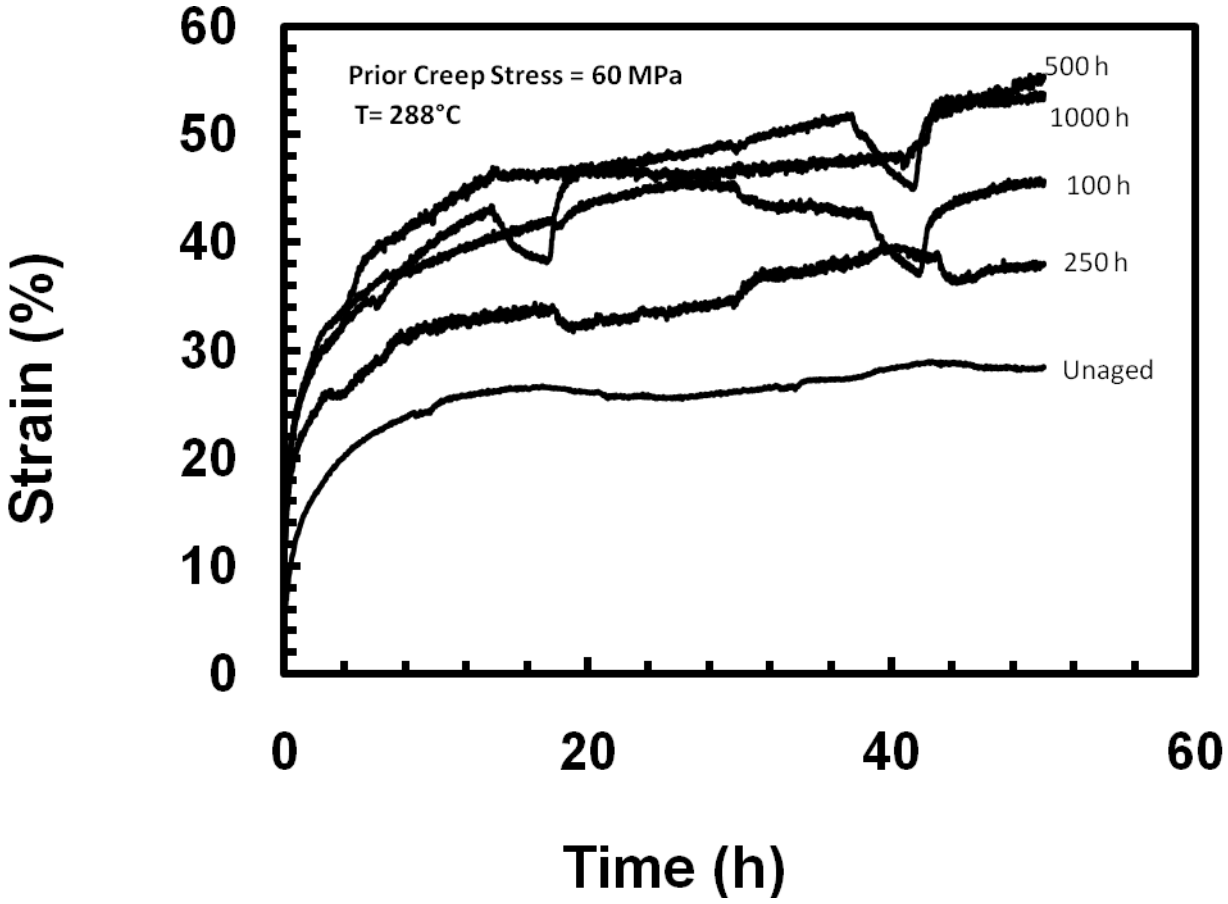


**Figure 48 Creep recovery as a function of recovery time for carbon fiber reinforced PMR-15 composite aged in argon at 288°C with a prior creep stress of 60 MPa**

Figure 48 shows the creep recovery as a function of recovery time. Interpretation of this result shows no particular trend to the data. The amount of prior aging time does not seem to be a significant influence on recovery at zero stress. This is contrary to the apparent trend of the data in Figure 47, which seemed to hint at an inverse relationship between recovery strain and aging time. It is unclear if the recovery behavior is a function of the prior aging condition or the magnitude of the creep strain accumulated, or some combination of both. The graph depicting the response of the 30 MPa prior creep stress specimens is not shown due to the data being obscured by electrical noise.

# Concluding Remarks

## Overview

This section illustrates the conclusions drawn from the results of this research.

## Remarks

Carbon fiber reinforced PMR-15 composite with an 8-harness satin fabric weave ±45 fiber orientation was aged in argon at 288°C for 100, 250, 500, and 1000 hours and compared to unaged specimens. Tensile and creep testing was conducted on each age group at 288°C. A creep stress of 30 MPa and 60 MPa was applied to the specimens for the duration of 25 hours, immediately followed by a recovery period of 50 hours. Weight loss as a function of time was taken for two rectangular specimens in parallel with the aged specimens. Tensile tests to failure were conducted to establish the Ultimate Tensile Strength for each age group. These specimens were then used to study the amount of oxidation layer growth. The elastic modulus was measured before and after aging.

The results of the creep tests make it clear that there is a direct relationship between the amount of aging time and the creep strain accumulation. The unaged specimens accumulated a creep strain of ~.72% at 30 MPa and ~1.86% at 60 MPa. The accumulated creep strain of the 1000 hour aged test specimen was ~.15% at 30 MPa and ~.91% at 60 MPa. It is clear from this data that lower creep stress causes lower creep strain. As mentioned previously, the PMR-15 neat resin showed an opposite reaction to increased aging time. The creep strain of PMR-15 neat resin increased the longer the specimen was aged [6].

The weight loss of the specimens was ~.91% at 250 hours and ~1.37% after 500 hours. The initial rapid weight loss was followed by a steady decrease in weight. This is an expected result as the weight loss in argon is dependent upon the volume of the specimen. The weight loss reaction differs in oxygen and in an inert environment because the weight loss is occurring evenly throughout the material. Oxidation degradation is not a factor, and thus the specimens showed no signs of oxidation layer growth.

The elastic modulus of each specimen was taken before any testing to establish the specimen-to-specimen variance. The average elastic modulus was determined to be 15.684 GPa with a covariance of .0817. The elastic modulus of loading and unloading was also taken during each test. The initial modulus was found to be higher than the aged moduli because of the damage accrued by the aging process. The modulus upon unloading was found to be lower than the modulus upon loading and is accredited to the damage sustained during the creep testing.

The effectiveness of this study is dependent upon the data gathered via creep testing. Many factors could have influenced the results of these individual tests, which is why performing the same tests multiple times can ensure accuracy of results. This would allow statistical modeling of the response and highlight any errors found in individual test specimens. Further exploration of the creep response, by extending the creep time, could also yield results that are not in line with results gathered for the 25 hour period of this research. The recovery period might be extended to ascertain whether the material is able to fully recover given enough time. Dynamic Modulus Analysis (DMA) could be conducted

to confirm changes to the glass transition temperature in order to see the changes in the crosslink density.

This thesis research was conducted in order to further expand current knowledge on the mechanical response of carbon fiber reinforced PMR-15 under sustained loading at elevated temperature. Research of this nature will hopefully allow for greater use of this composite in the aeronautical and aerospace industry.

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## Appendix A

## Appendix A: Fracture and Oxidation growth micrographs for test specimens subjected to tensile testing at 288°C

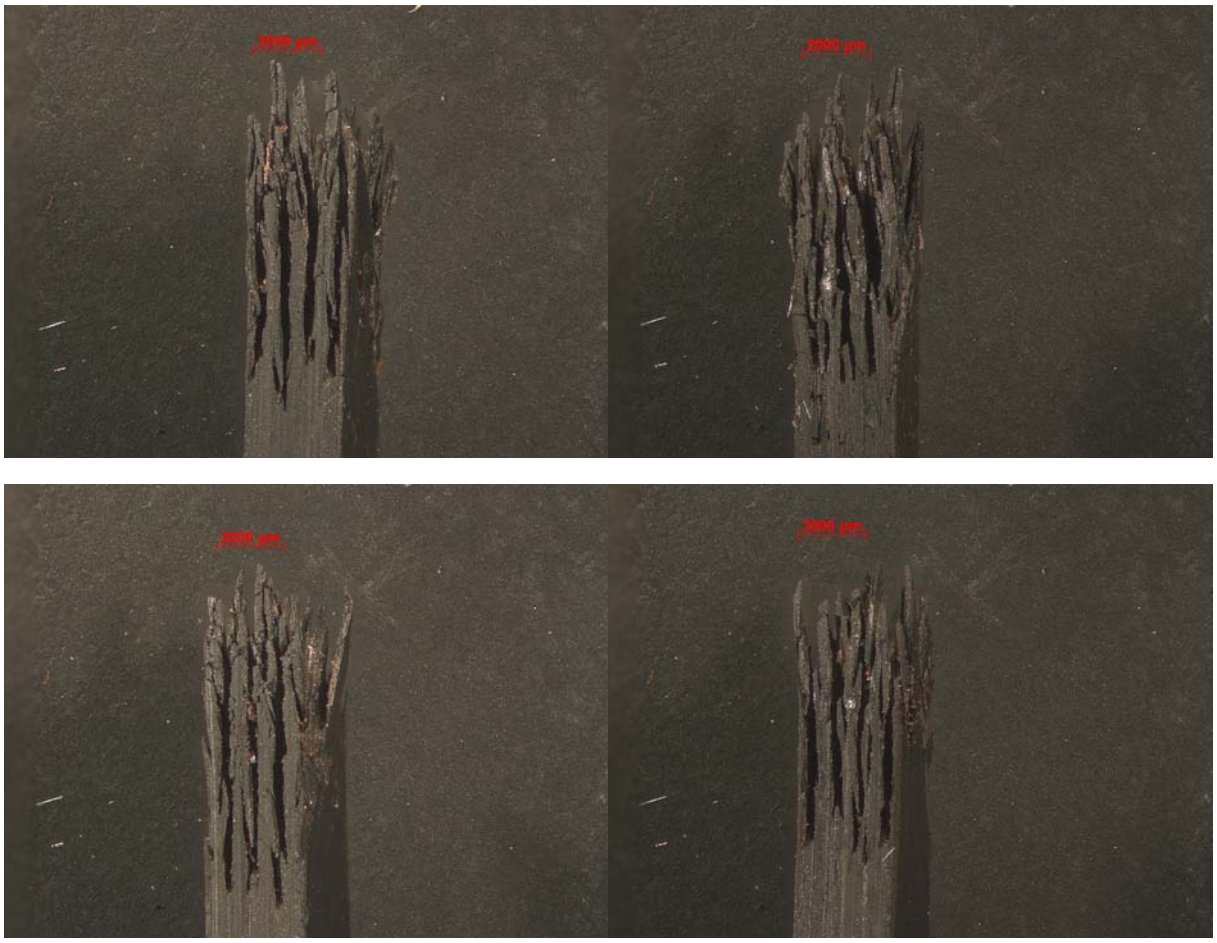

**Figure 49 Carbon fiber reinforced PMR-15 composite unaged specimen 21 (side view).**

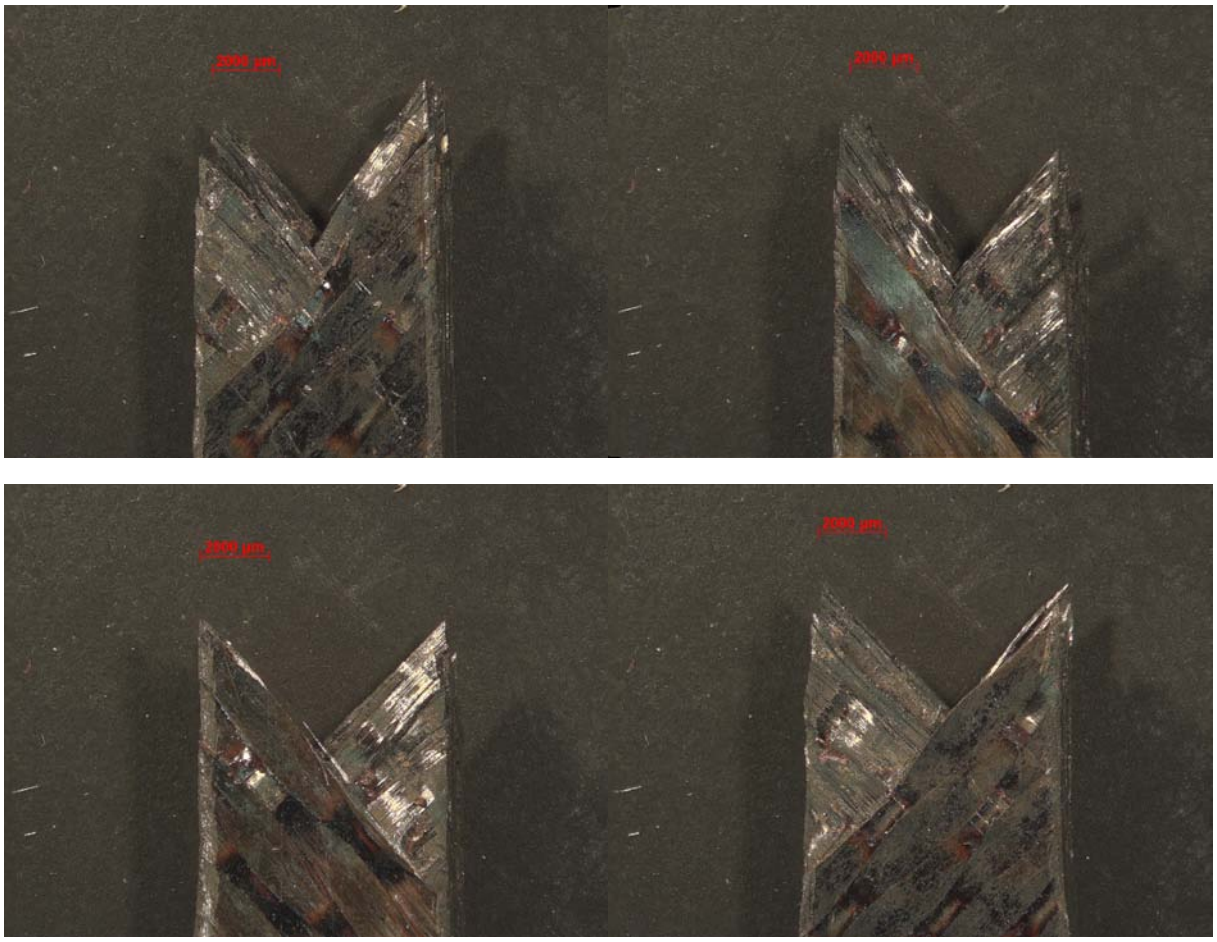

**Figure 50 Carbon fiber reinforced PMR-15 composite unaged specimen 21 (top view).**

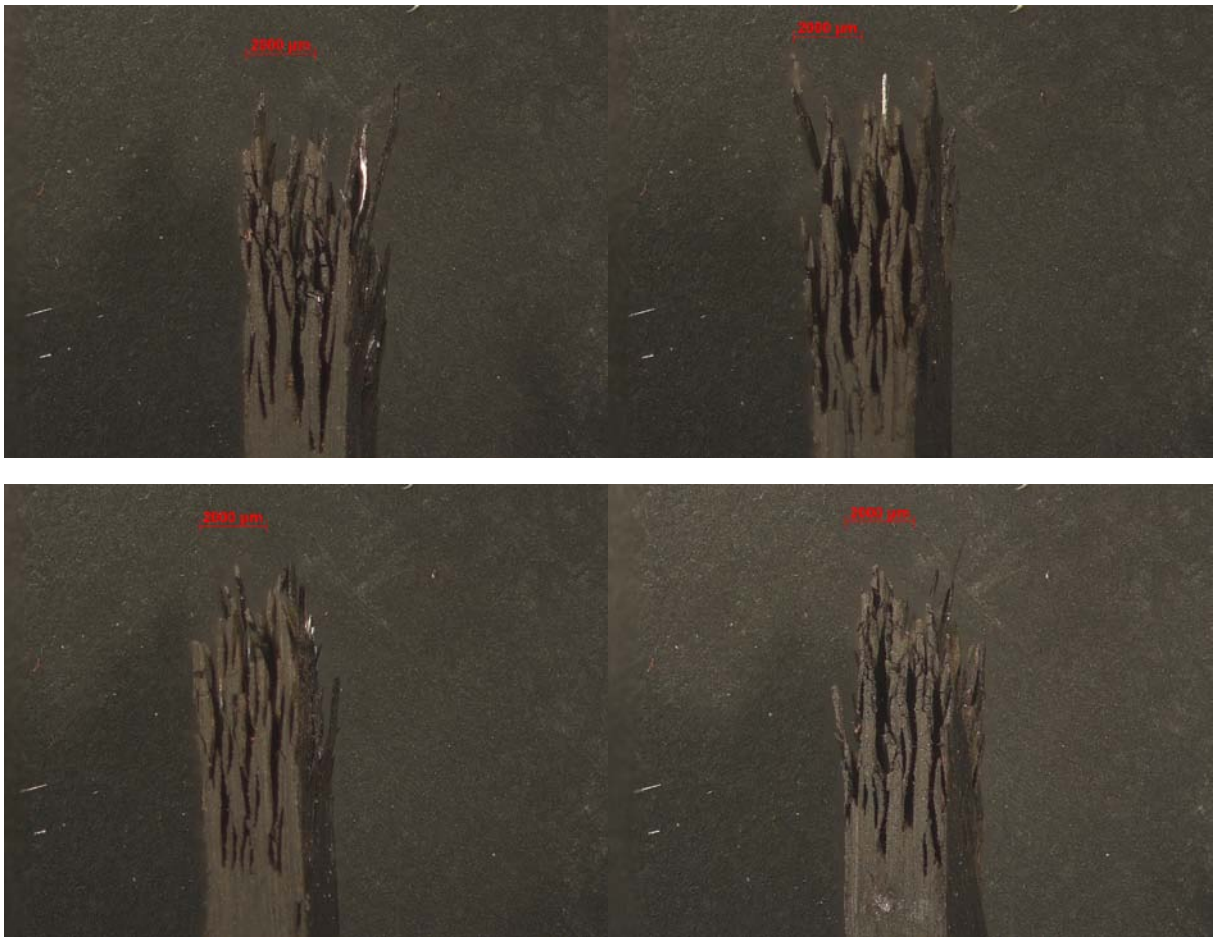


**Figure 51 Carbon fiber reinforced PMR-15 composite specimen 2 aged in argon for 100 hours at 288°C (side view).**

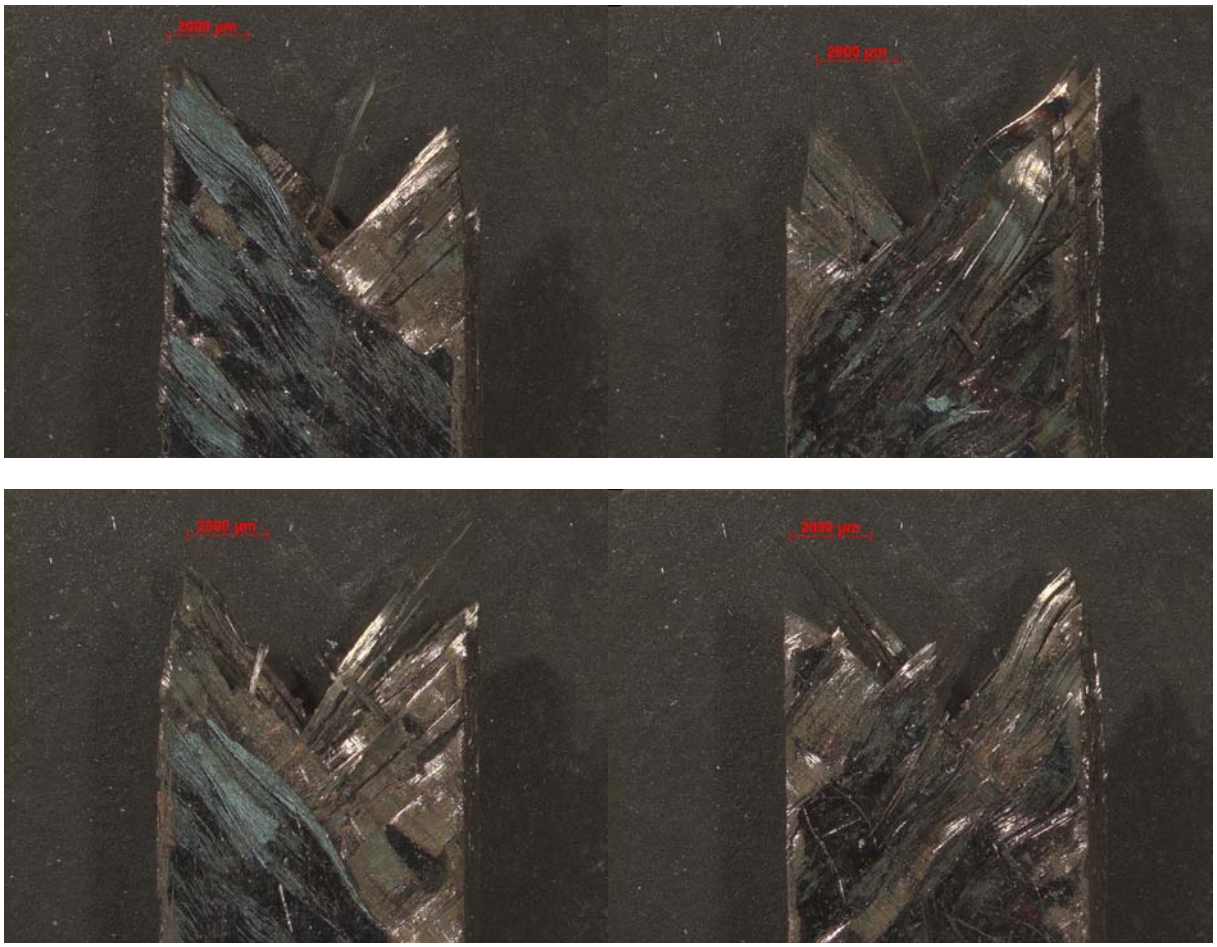


**Figure 52 Carbon fiber reinforced PMR-15 composite specimen 2 aged in argon for 100 hours at 288°C (top view).**

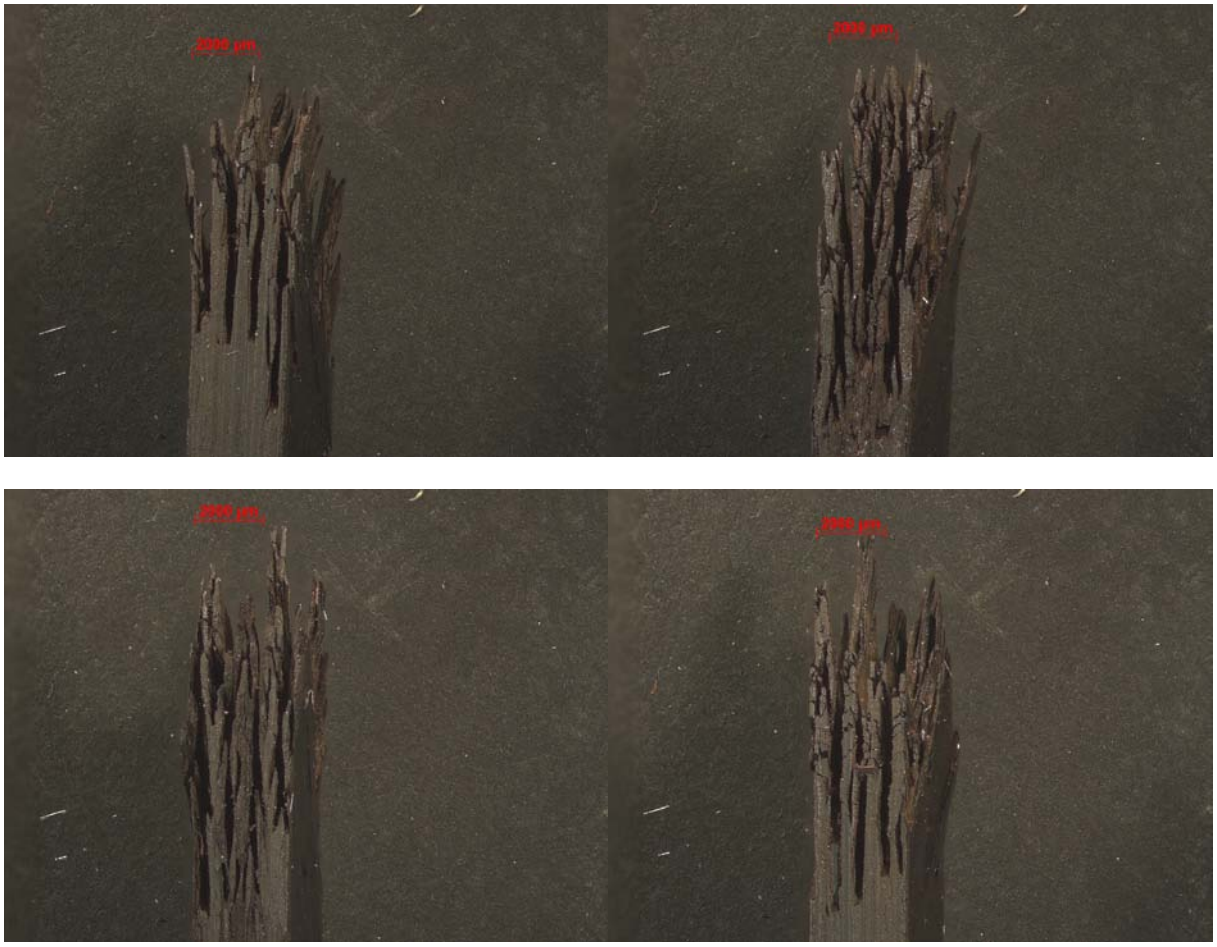

**Figure 53 Carbon fiber reinforced PMR-15 composite specimen 16 aged in argon for 250 hours at 288°C (side view).**

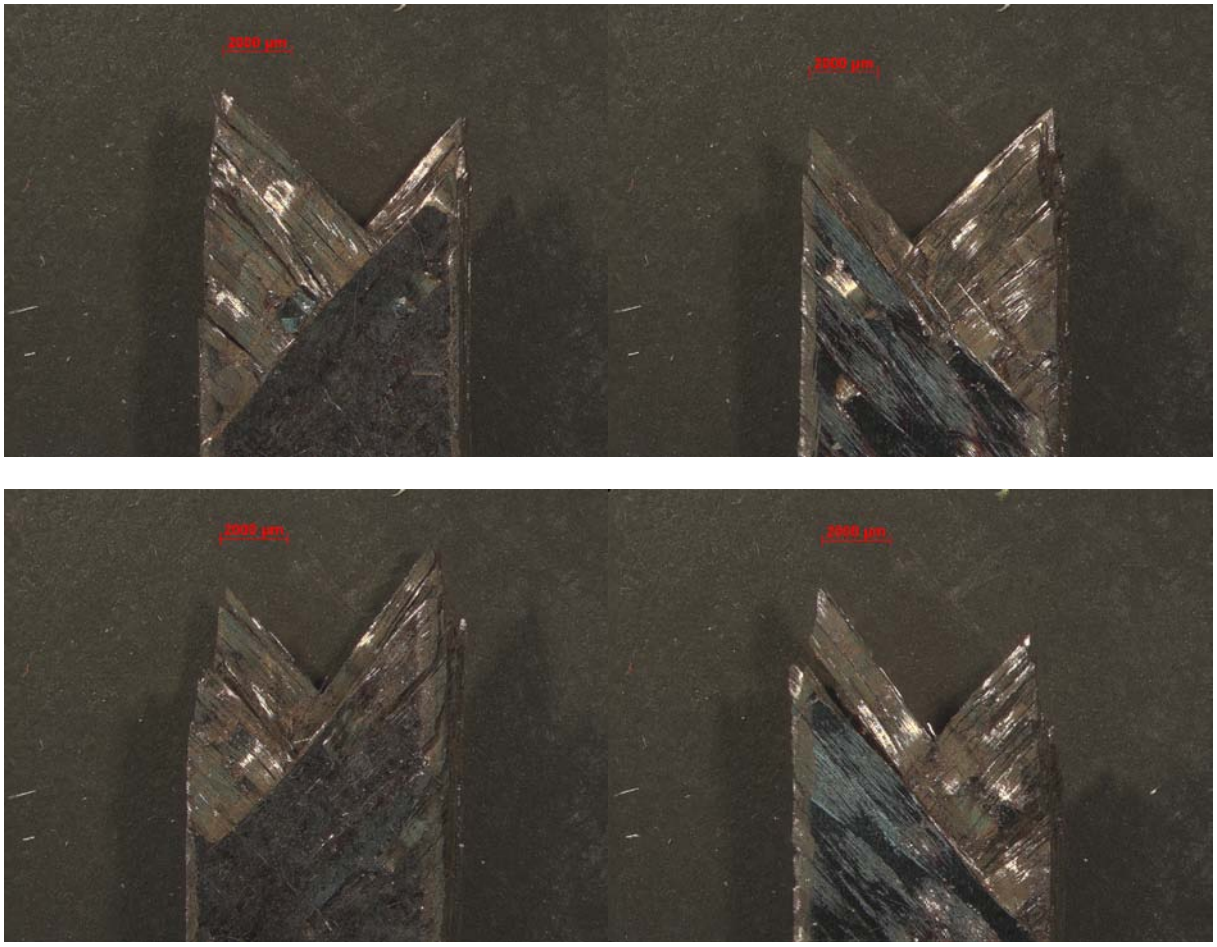

**Figure 54 Carbon fiber reinforced PMR-15 composite specimen 16 aged in argon for 250 hours at 288°C (top view).**

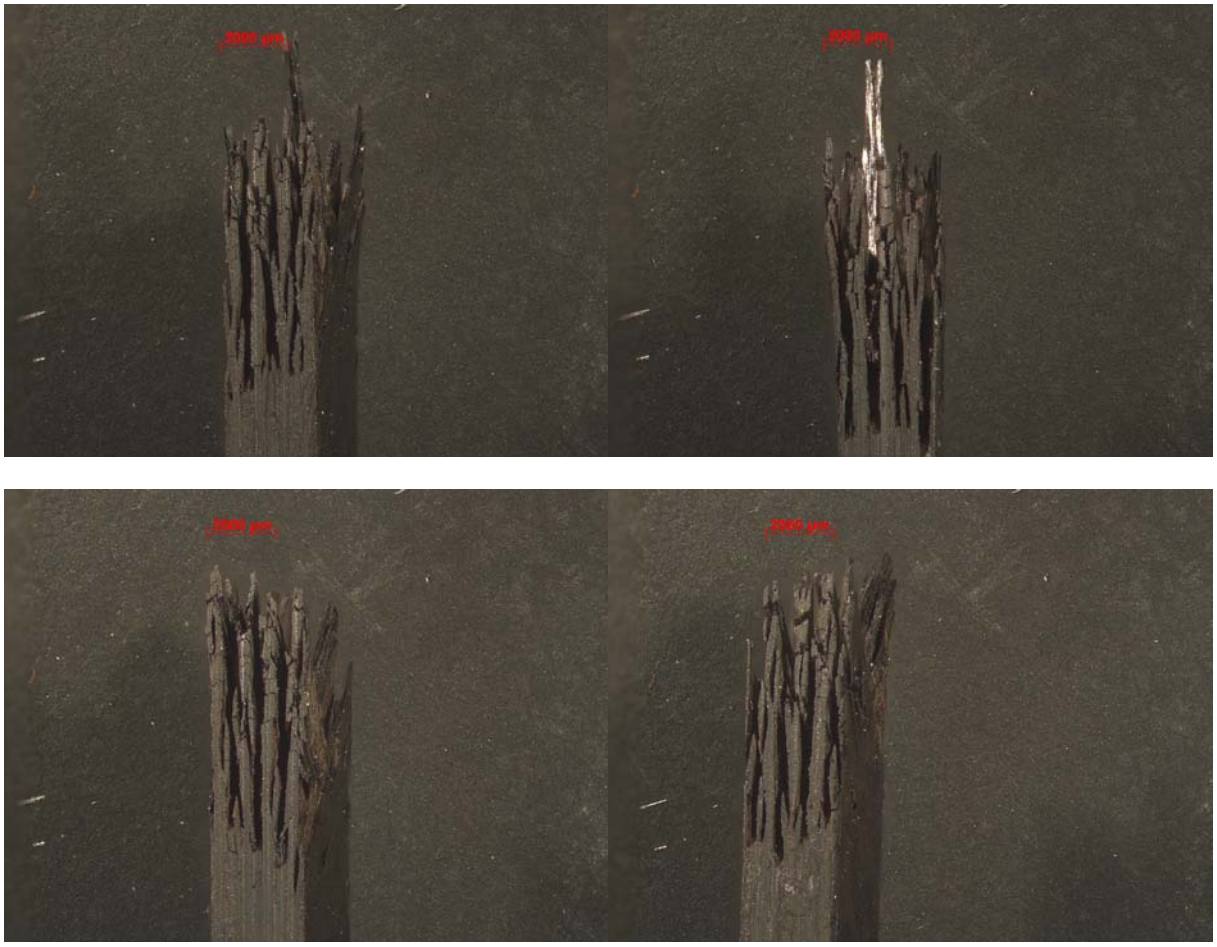

**Figure 55 Carbon fiber reinforced PMR-15 composite specimen 6 aged in argon for 500 hours at 288°C (side view).**

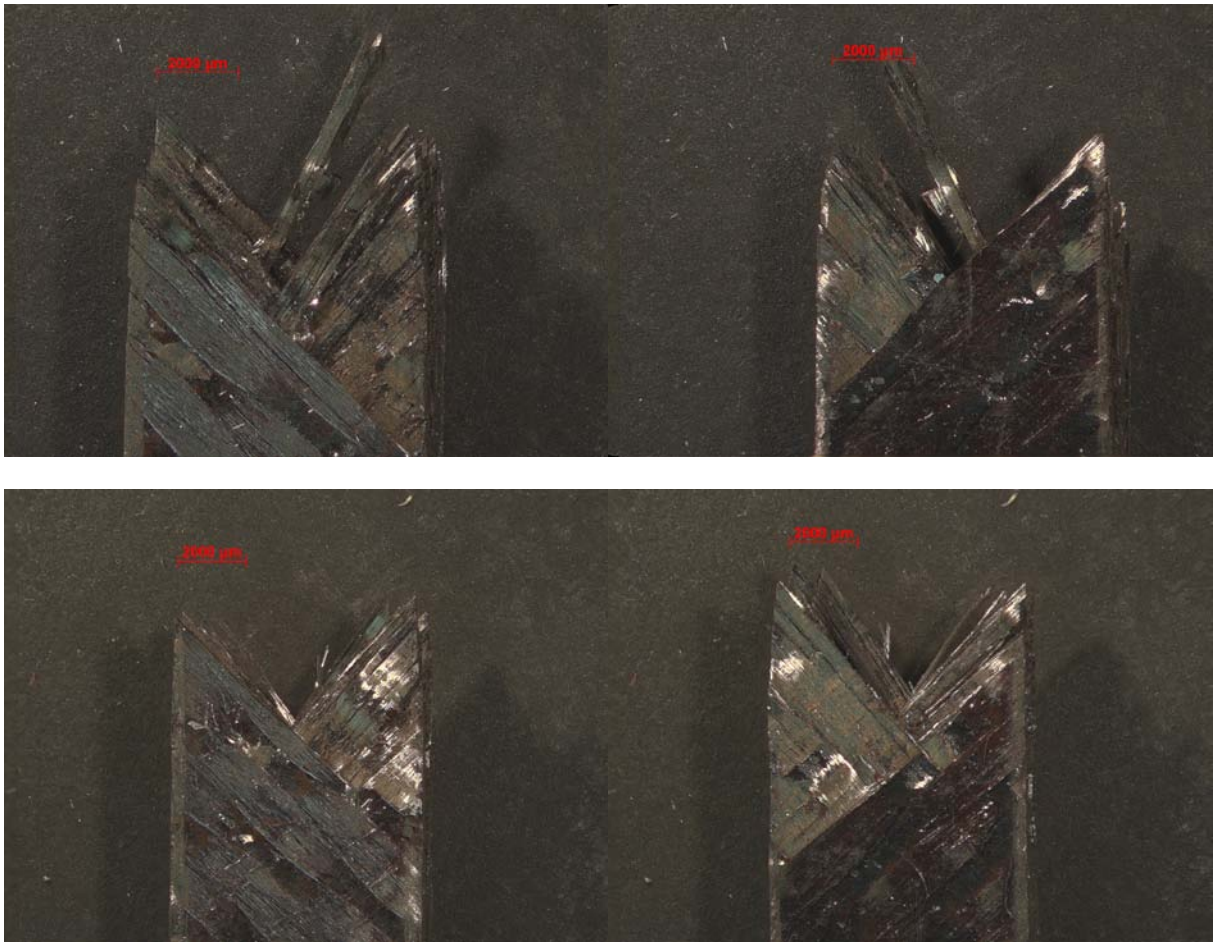

**Figure 56 Carbon fiber reinforced PMR-15 composite specimen 6 aged in argon for 500 hours at 288°C (top view).**

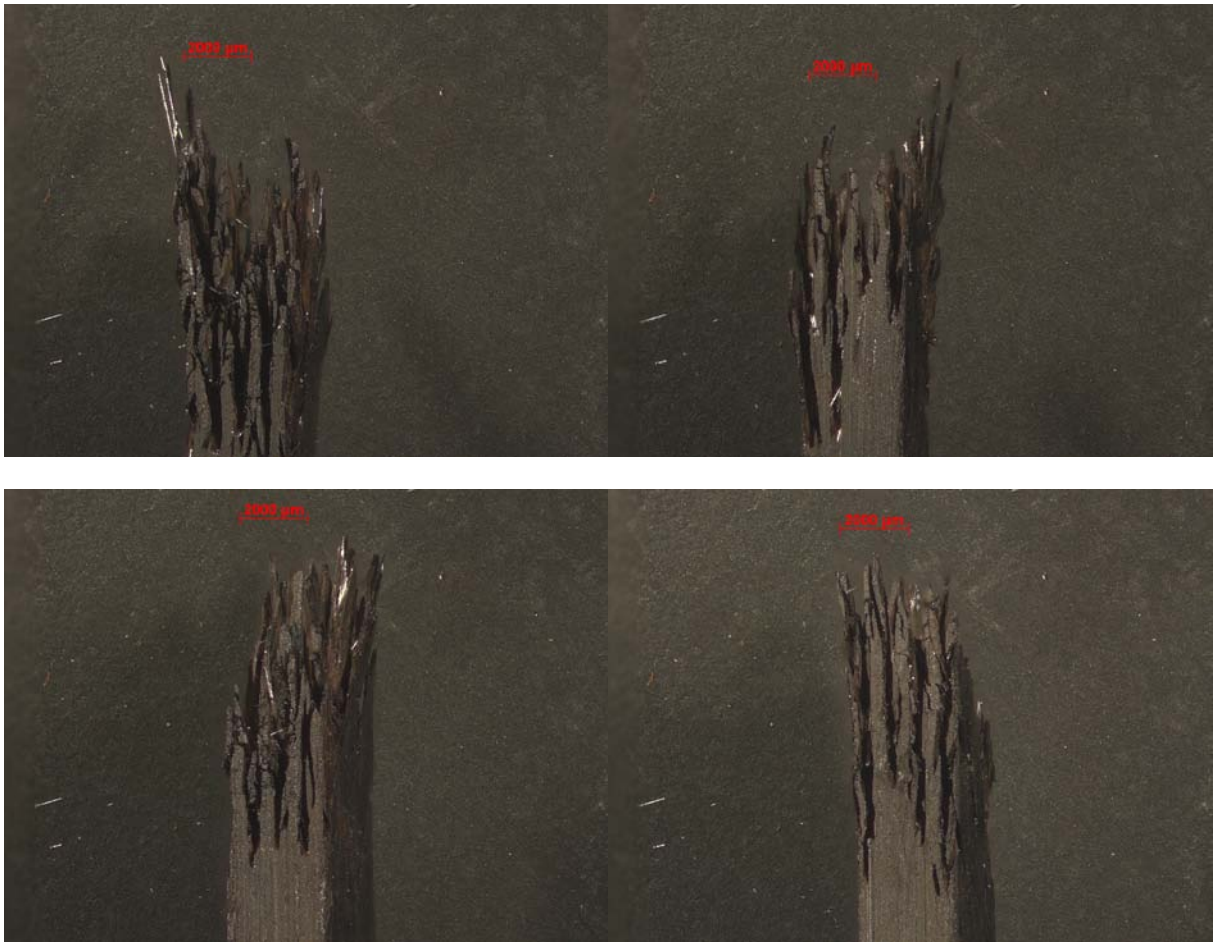

**Figure 57 Carbon fiber reinforce PMR-15 composite specimen 11 aged in argon for 1000 hours at 288°C (side view).**

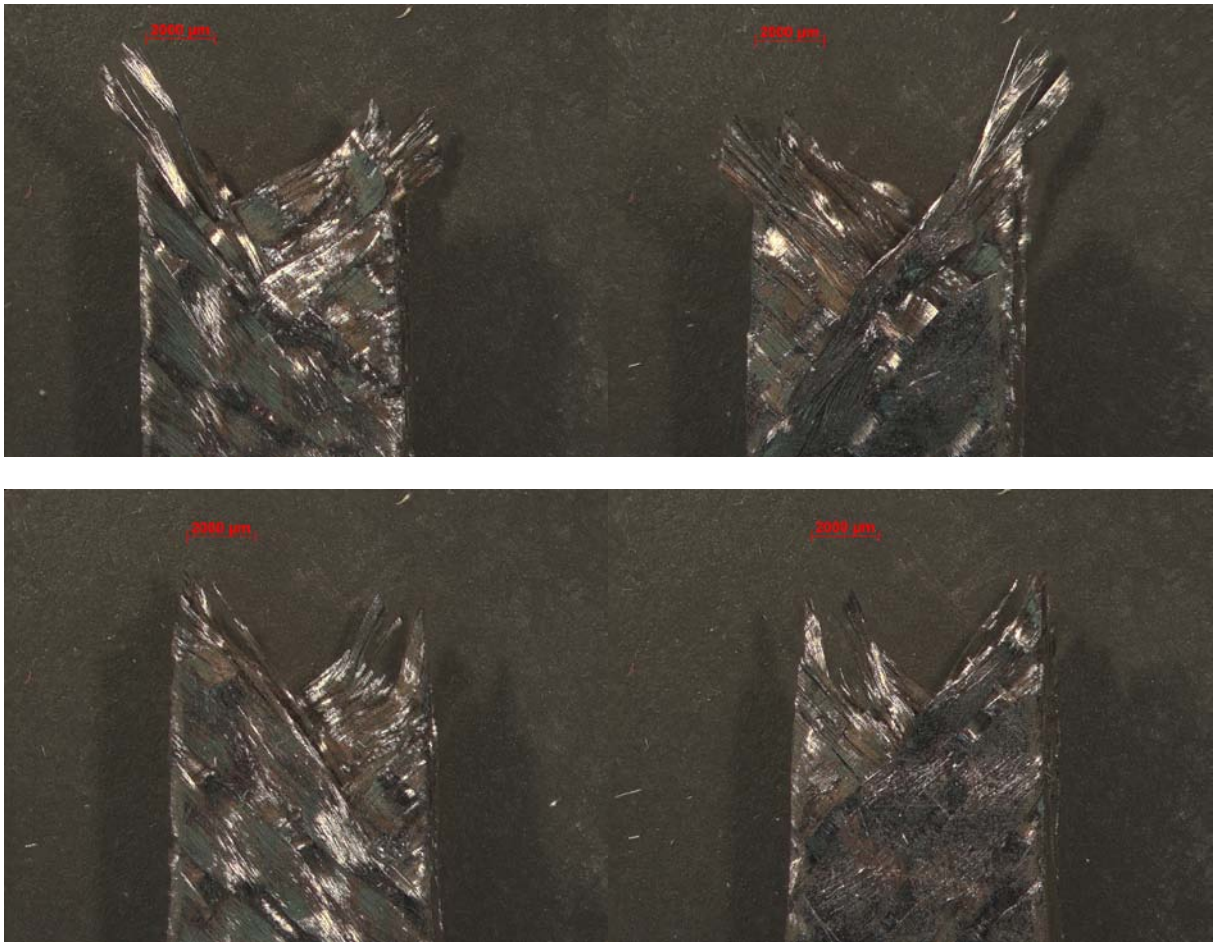

**Figure 58 Carbon fiber reinforced PMR-15 composite specimen 11 aged in argon for 1000 hours at 288°C (top view).**

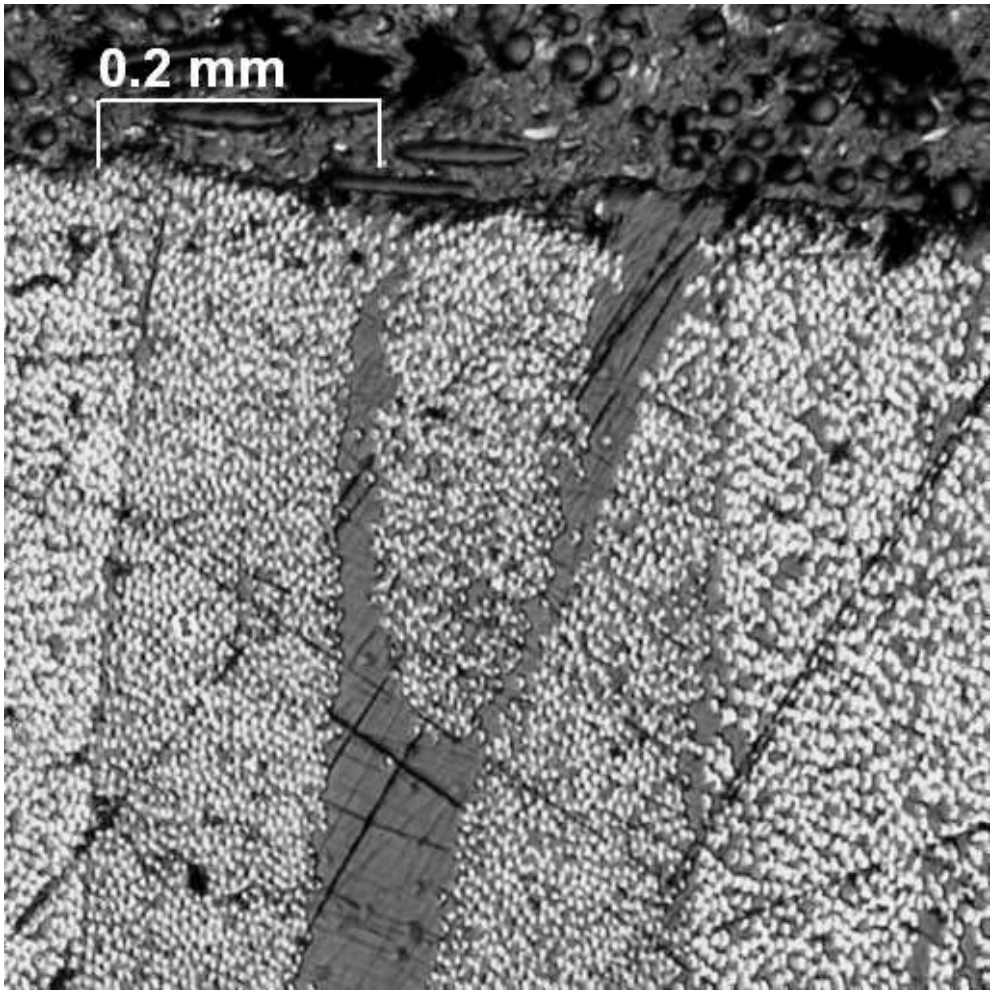


**Figure 59 No oxidation layer observed on polished section of unaged specimen.**

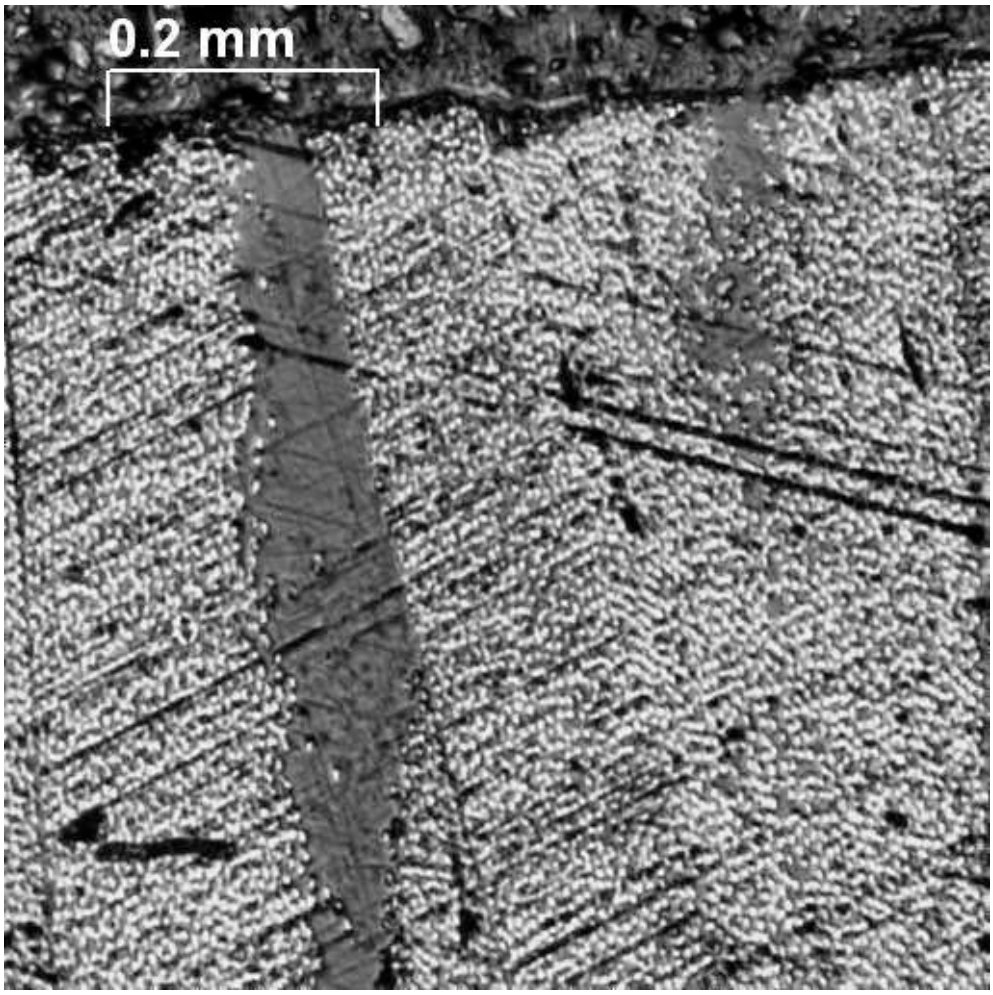


**Figure 60 No oxidation layer observed on polished section of sample aged in argon at 288°C for 100 hours.**

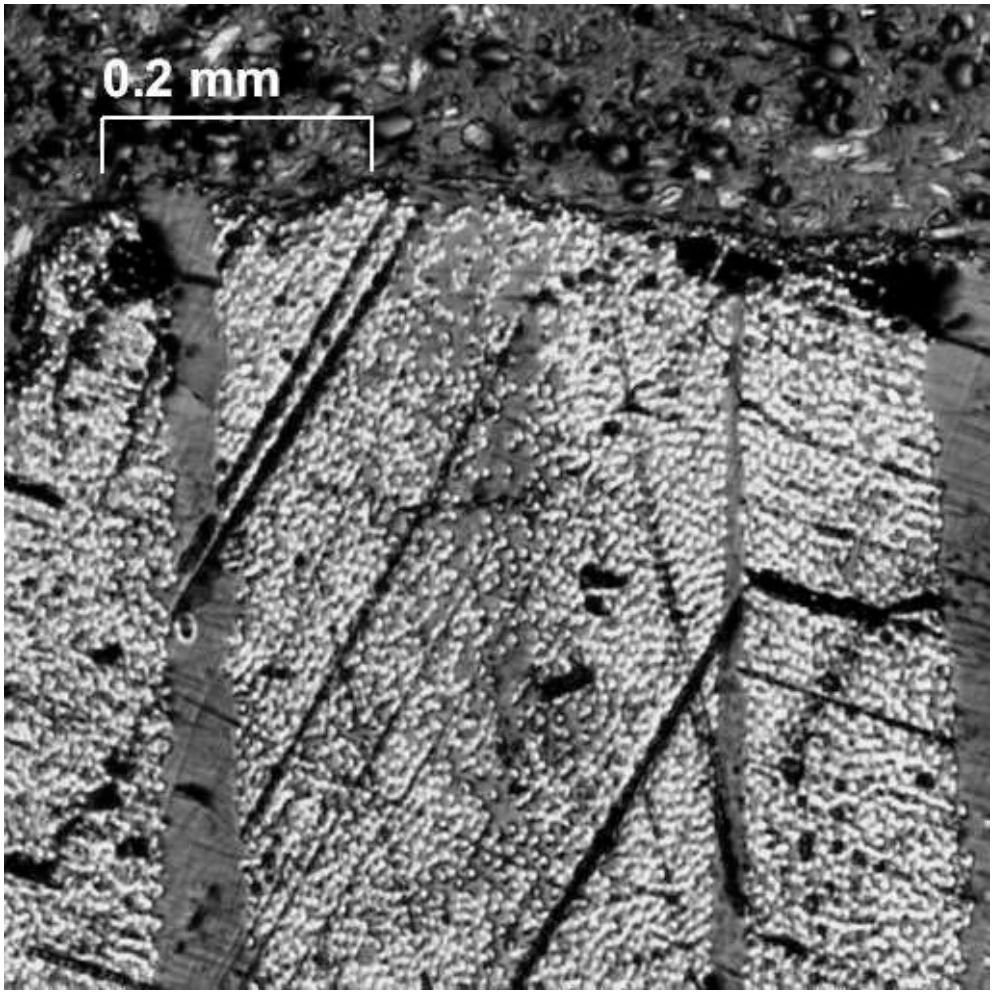


**Figure 61 No oxidation layer observed on polished section of sample aged in argon at 288°C for 250 hours.**

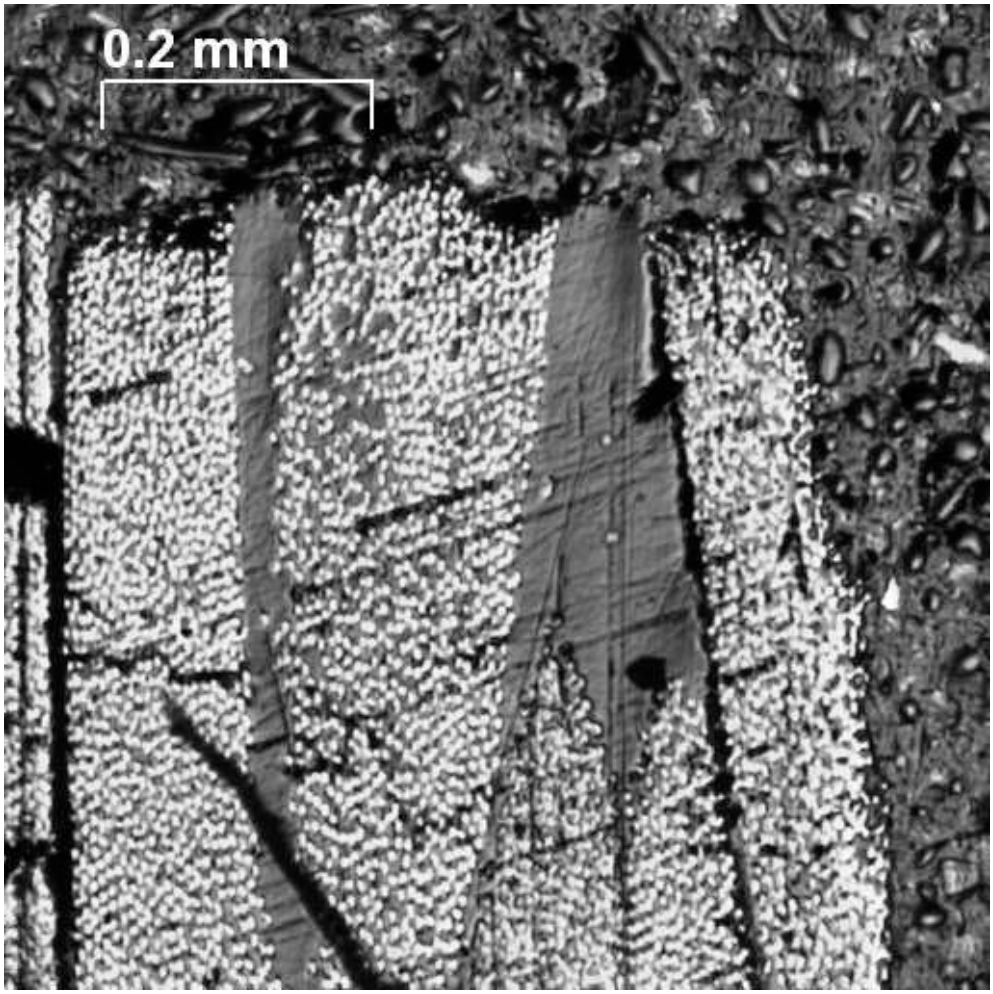


**Figure 62 No oxidation layer observed on polished section of sample aged in argon at 288°C for 500 hours.**

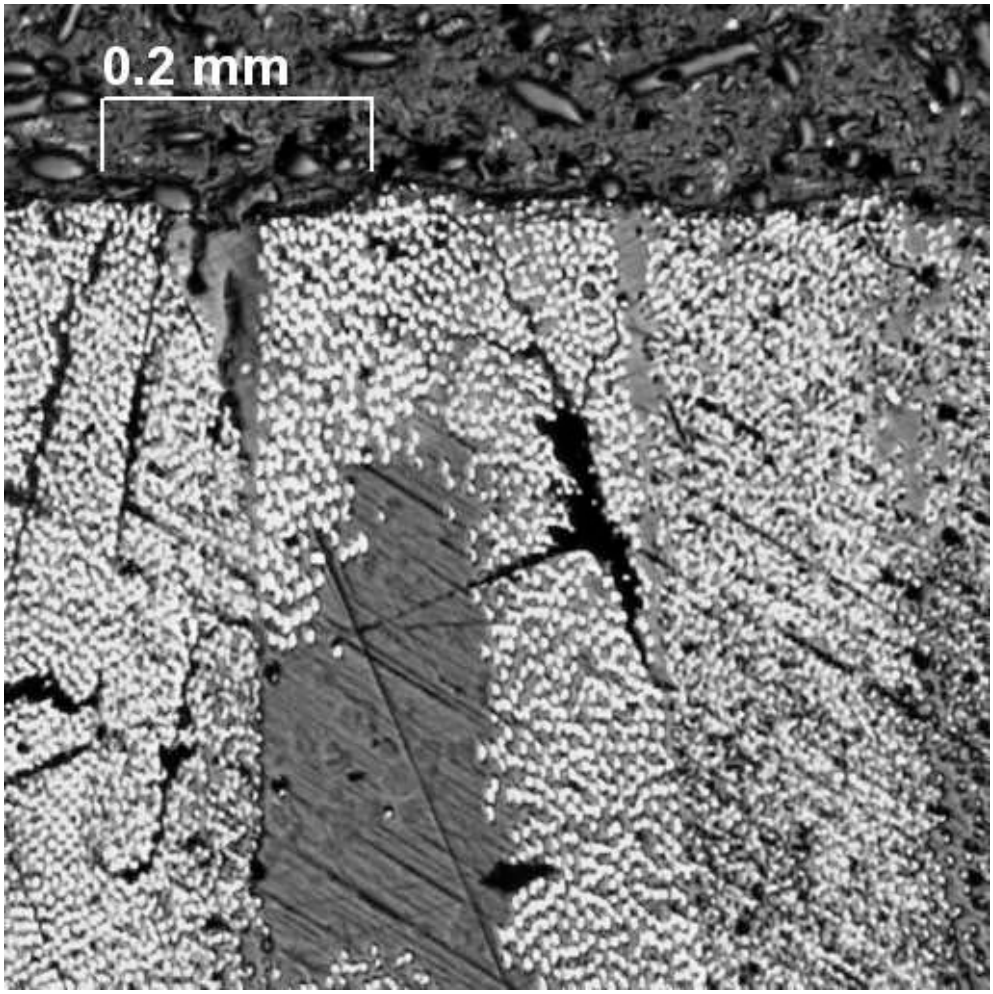


**Figure 63 No oxidation layer observed on polished section of sample aged in argon at 288°C for 1000 hours.**

## Vita

Tyler Gruters graduated from Minster High School in Minster, OH. He entered undergraduate studies at Franciscan University of Steubenville in Steubenville, OH where he graduated with a Bachelor of Science in Engineering, Mathematics, and Philosophy. He continued his education at the Air force Institute of Technology where he will be graduating with a Masters Degree of Aeronautical Engineering in June 2009. He concurrently attended Wright State University and expects to graduate with a Masters Degree of Mechanical Engineering in December 2009.

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